METALLURGIA

THE BRITISH JOURNAL OF METALS

Vol. 62 No. 373 NOVEMBER, 1960 Monthly: Two Shillings and Sixpence

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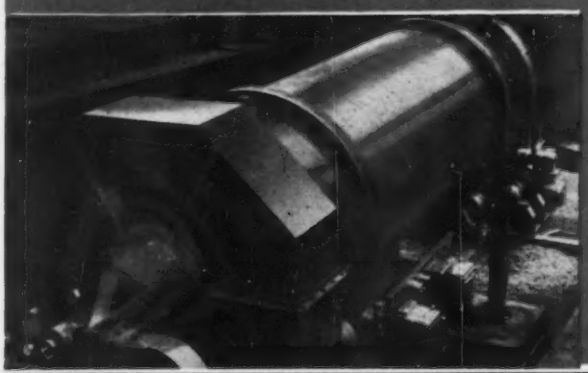


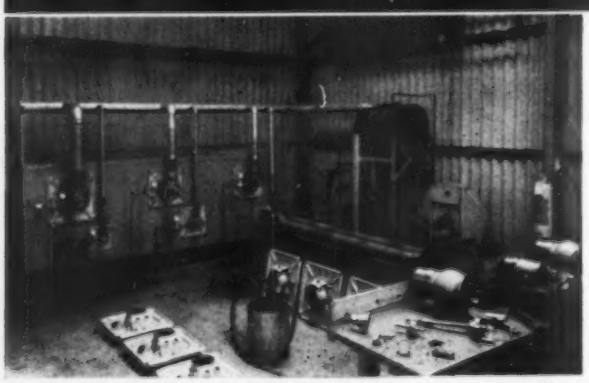
ASPHALT PLANT

A Stordy-Hauck self-proportioning oil burner applied to a rotary dryer and asphalt plant.

This burner more than meets the operating temperature range required for varying materials handled.

GLASS MELTING

Stordy-Hauck self-proportioning oil burners are fitted to this Hartford Empire Unit glass melting installation, comprising 18 burners all under full automatic control.



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Stordy manufacture at their Wombourn Works a full range of Hauck fully self-proportioning oil burners with full facilities for experimental work and for full testing of production burners before despatch.

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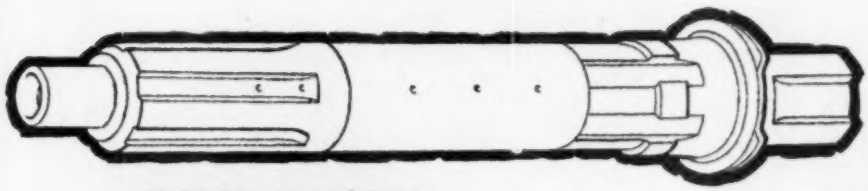

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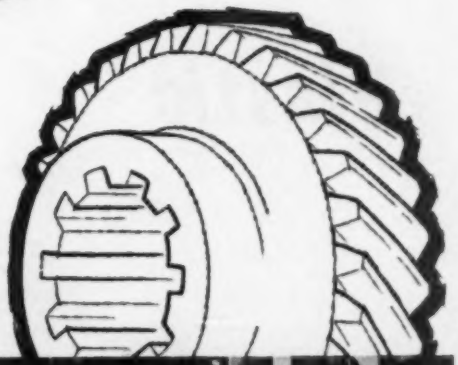

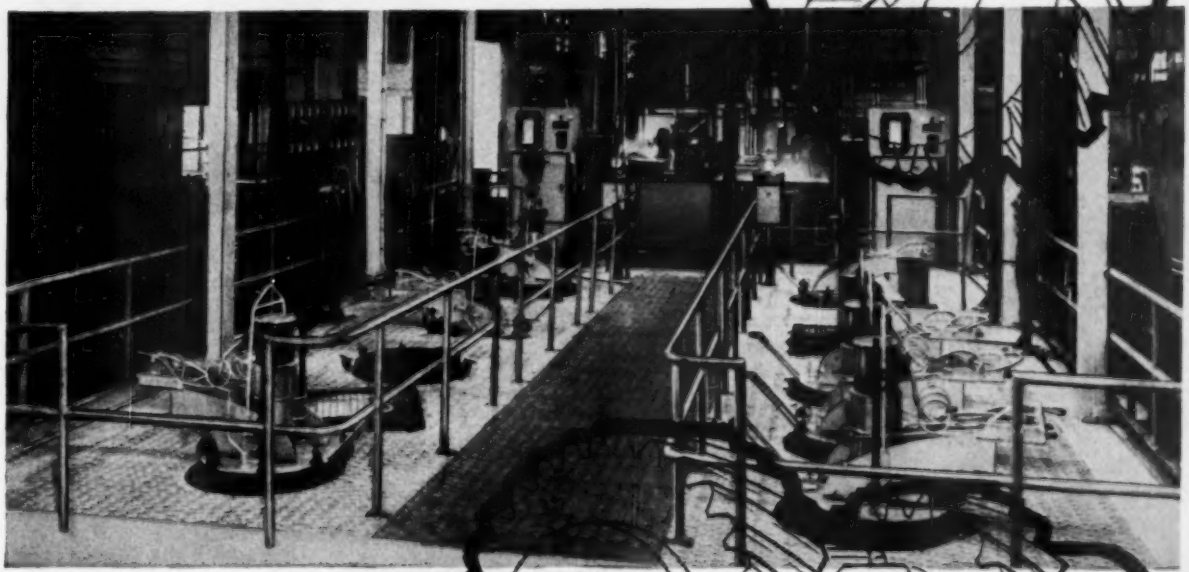

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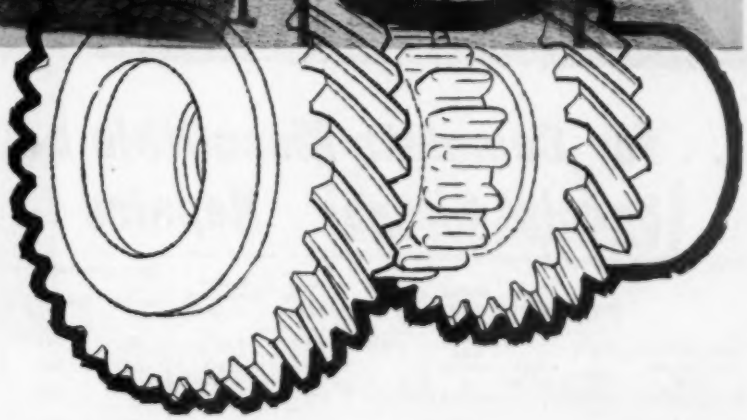

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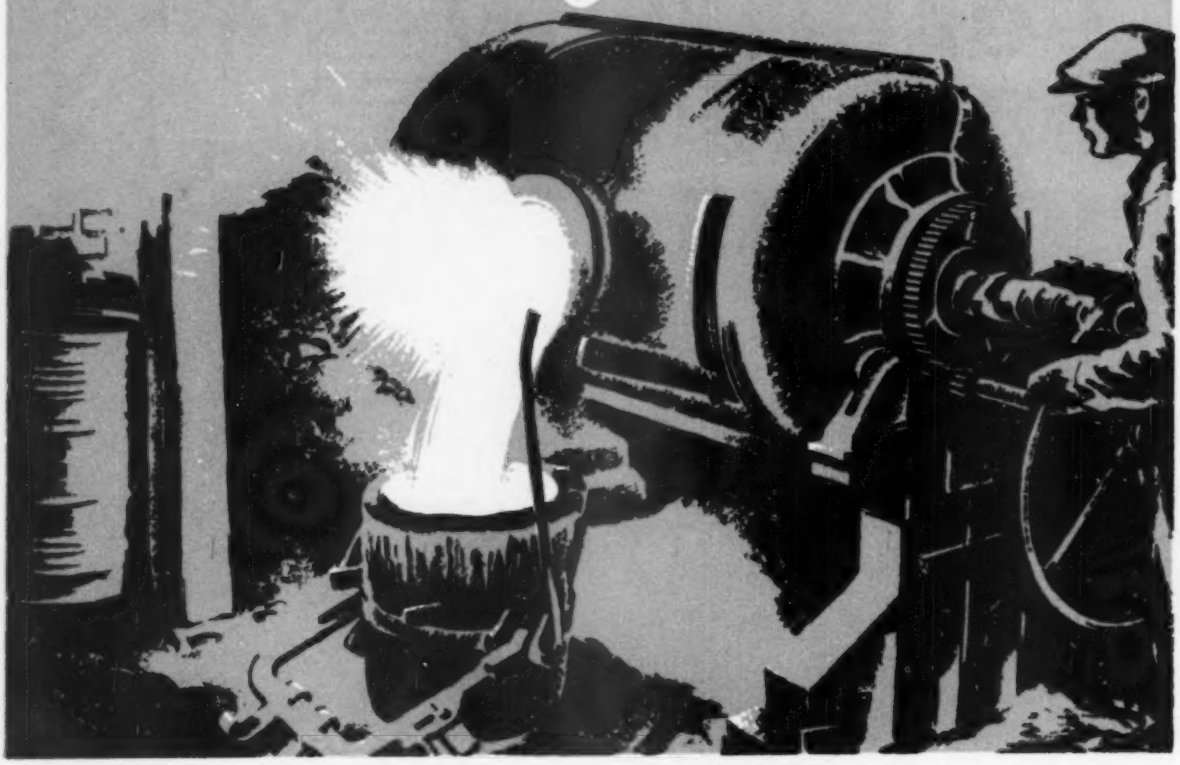
WB92

STEIN *Refractories*

Plastic



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	How Supplied	Type of Set	Methods of Use	Refractoriness	Thermal Hardening Temperature	Maximum Temperature of use	Approx. weight required per cubic foot lbs.	Approx. weight required per cubic metre kgs.
Moksicoar Plastic	Plastic	Heat Setting	Hand moulding or patching	1730°C	900°C	1580°C	140	2243
Stein Sillimanite Patch	Plastic				1000°C	1760°C	155	2483
Stein 73 Plastic	Plastic			+ 1750°C	1000°C	1800°C	180	2884
Moksicoar Fire Putty	Plastic	Air Setting	Hand moulding or patching	1350°C	500°C	1100°C	140	2243
Stein Carome Patch	Semi-Plastic			+ 1750°C	600°C	1500°C	200	3204
Moksicoar Patch	Plastic		Ramming or patching	1600°C	600°C	1550°C	135	2162
Stein 73 Patch	Semi-Plastic			+ 1750°C	600°C	1800°C	175	2804
Stein Magnesite Ramming Mixture	Dry			+ 1750°C	1400°C	1750°C	175	2804

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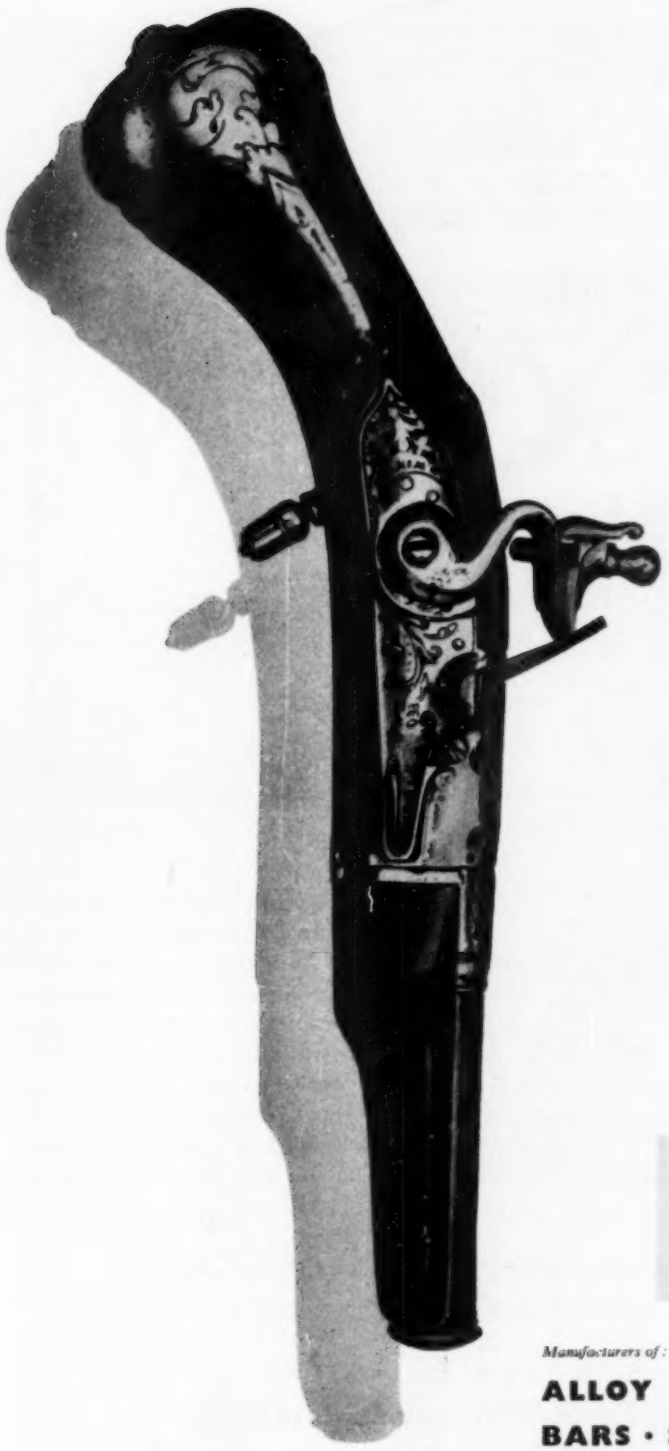

Characteristic of British Craftsmanship is this handsomely worked pistol. Made in the 16th Century from steel and wood it is a fine example of the work of that period and can be seen in the Victoria and Albert Museum.

Britain is proud of her craftsmen ; throughout the years their skilled workmanship has earned a reputation for Britain that is second to none. The historical associations of steel in England chronicled through the ages, show how dependent we have become upon its production. Today its manifold uses in industry and everyday life have become legion—civilisation itself is woven over a framework of iron and steel. Hallamshire high grade Alloy and Carbon steels are an integral part of Britain's traditional good name.

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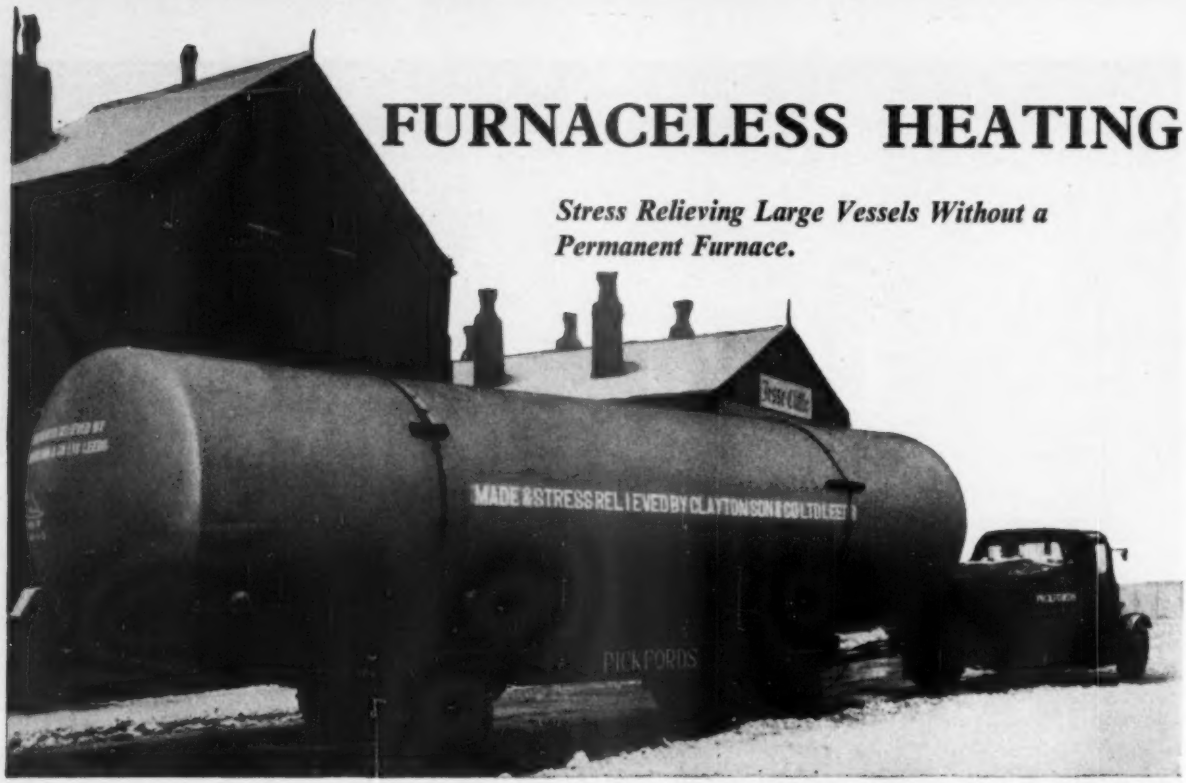

Fig. 1. 46′ × 9′ diameter high pressure cylinder, stress relieved *in situ*.

Clayton Son & Co. Ltd., of Leeds, have been well known as heavy engineers for nearly 100 years and one important aspect of their business is the fabrication of large welded pressure vessels. Recently they received an order for two high pressure propane gas cylinders, one of which is shown in Figure 1. These are of all-welded construction and to comply with British Standard 1500, dealing with stress relieving of pressure vessels after welding, the whole of the vessel must be heated after fabrication according to an extremely exact time/temperature schedule. When the stress relieving temperature of the vessel is at 650°C, the temperature differential between any two points must not exceed 50°C.

The usual method of meeting these requirements is to place the pressure vessel in a gas-heated furnace and raise its temperature in accordance with the requirements of B.S. 1500. However, as the dimensions of the vessels increased — the gas cylinders mentioned above being 46′ x 9′ in diameter—the number of stress relieving furnaces available for this class of work was limited. Sometimes the job had to be done by inserting one end of the container into the furnace with the other end protruding, and then reversing it. This was obviously uneconomical in terms of fuel and hardly met the real requirements of B.S. 1500.

Messrs. Claytons discussed their problem with the Industrial Development Centre of the North Eastern Gas Board, and they undertook experiments with high velocity jet type gas burners, working on an old Lancashire boiler shell. Within six months, the North Eastern Gas Board had evolved a method of stress relieving these larger containers *in situ*, obviating the inconvenience and expense of transporting the vessel to another factory for the stress relieving to be carried out. This stress relieving process is covered by a joint patent held by the North Eastern Gas Board and Clayton Son & Co. Ltd.

A model of the equipment used is shown in Figure 3 and the burner is shown in Figure 2.

First the whole of the vessel was lagged with an insulating material which in this case was 4″ of Rocksil. The jet burner was then arranged to fire through the manhole of the vessel into a hot gas distribution tube arranged internally, having a number of perforations, as shown in Figure 4. The hot blast of gas through the central distribution tube causes, by entrainment, the recirculation of waste gases within the centre tube, distributing the heat uniformly throughout the vessel. In addition, arrangements were made for jets of compressed air to control the hot gas flow from the ends of the distribution tube to provide uniform heating at the ends of the vessel. Finally, the hot gases escape through the same vent through which the burner fires, as shown in Figure 4. Since it is necessary to record the temperature of the vessel at points not more than 15 feet centres, steel blocks were welded to the vessel and carried the thermocouple wires. A continuous record of the vessel temperature was main-

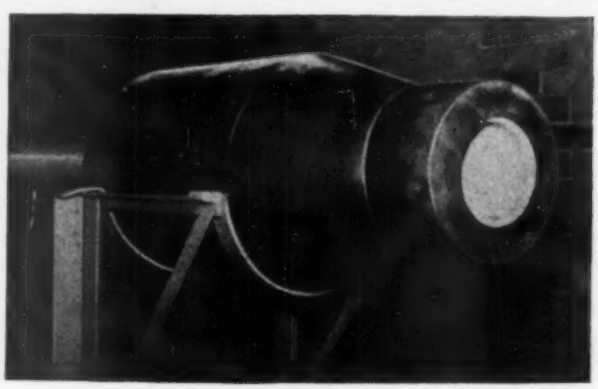

Fig. 2.
High velocity jet type gas burner.

tained over the heating and cooling periods and experience seems to indicate that the temperature schedule can be maintained even more accurately than is at present required by the B.S. Actual records obtained are shown in Figure 5. The following data give some idea of the performance achieved in the stress relieving of the high pressure cylinder shown in Figure 1.

Dimensions of Vessel

Length.....Approximately 46 ft.
Diameter .....9 ft.
Plate thickness.....$\frac{7}{8}$" shell, 1" endplates
Access hole..18" diameter, 6 ft. from end
Weight.....Approximately 21 tons

Burner Dimensions

Overall length.....28"
Exit port .....4½"
Air supply pressure.....40"
Air capacity available.....1000 c.f.m.
Gas maximum capacity..7000 cu. ft. per hr.
Jet speed (tunnel exit)..Approx. 400 m.p.h.
Recirculating tube.....15" diameter
Discharge holes....3"—3½" dia. holes along its length with 6"—9" end discharge.

Operational Details

Normal heating rate..7 hrs. to reach 600°C
Average gas rate....4200 cu. ft. per hour

The thermocouple readings over the period under investigation are given in Figure 5 and the inspecting authorities showed a great interest in the process, which they thought to be an improvement over prevailing methods. It is of interest to note that the Rocksil lagging cost about £280 with an additional £75 for its application and the installation of the centre tube. It is thus obvious that stress relieving of large vessels can now be carried out at minimum capital cost by fabricators without furnaces, eliminating transport to furnace plant with freight charges estimated at no less than £1 per ton per mile.

This is, of course, only one practical application of furnaceless heating. The North Eastern Gas Board and Messrs. Clayton Son & Co. Ltd., are experimenting with the erection of a temporary furnace which might be likened to a form of Nissen hut in sheet metal. This is lagged with Rocksil and irregular shaped articles of large tonnage may be placed inside this canopy. Once again a central tube arrangement is used, the jet burner being so placed that the whole of the irregular shaped component may be uniformly heated. Such a furnace is to be built by Messrs. Claytons.

The North Eastern Gas Board is also using this for the drying out and heating up of large glass furnaces after they have been repaired or rebuilt. Here again is an instance where the time/temperature schedule must be rigorously controlled because of the various physical and chemical changes which occur in the furnace brick work during drying out and pre-heating periods. The summarised advantages of furnaceless heating include:

a Minimum capital cost.
b It is no longer necessary to occupy permanent factory space with a huge furnace.
c Reduced running costs.
d The advantages of stress relieving *in situ*.

The North Eastern Gas Board is convinced of the importance of the applications of the principle of high velocity jet type burners for many heat treatment processes without using a permanent furnace structure. Typical applications include pressure vessels for the chemical and oil refinery industries as well as those employed in nuclear energy projects. This is one example only of the manner in which the Industrial Development Centres of the Gas Boards undertake work on behalf of and in co-operation with manufacturers. Their experiences are interchanged so that any manufacturer approaching his Industrial Gas Engineer has the benefit of this co-ordinated knowledge of applied industrial gas techniques.

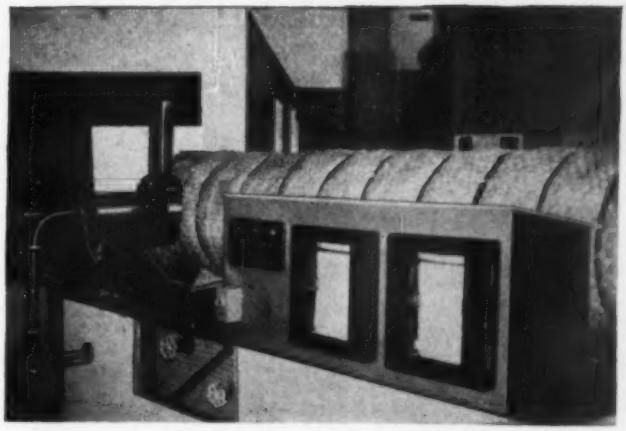

Fig. 3. Model of equipment used in furnaceless heating process.

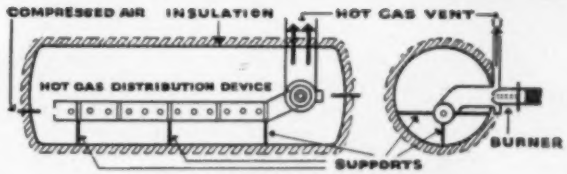

Fig. 4. Diagrammatic section through lagged cylinder showing burner jet and distribution tube.

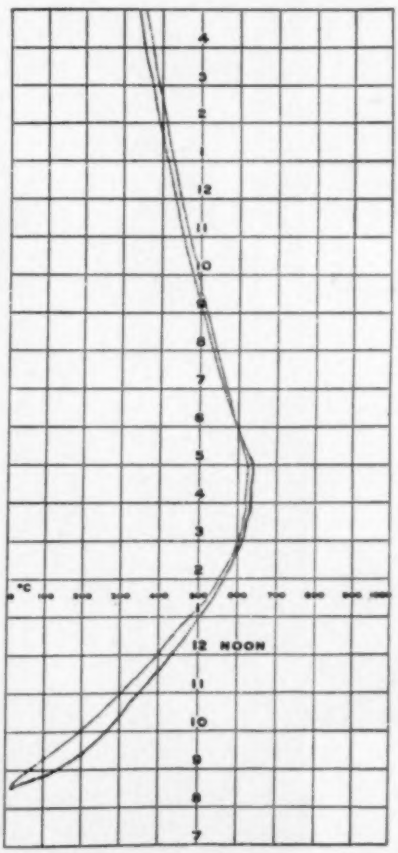

Fig. 5. Continuous temperature recording of heating and cooling process. (Maximum and minimum curves of the 12 points recorded).

Scottish Gas Board, Edinburgh
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North Western Gas Board, Manchester
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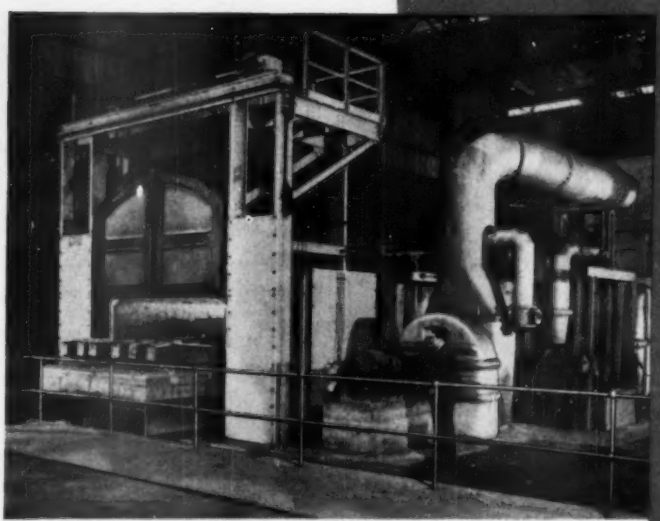

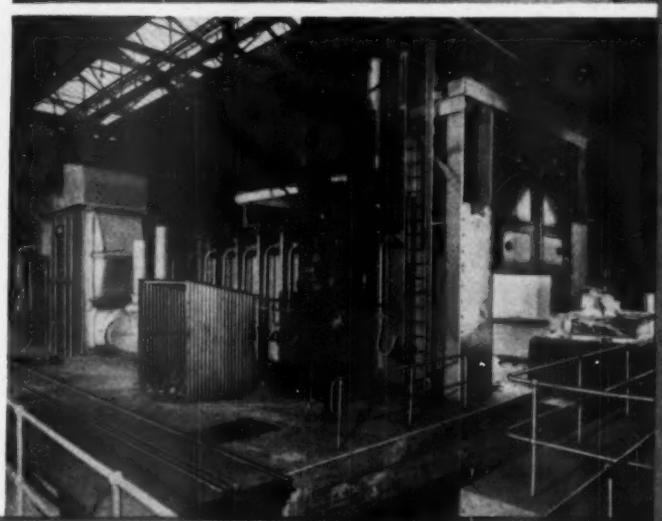

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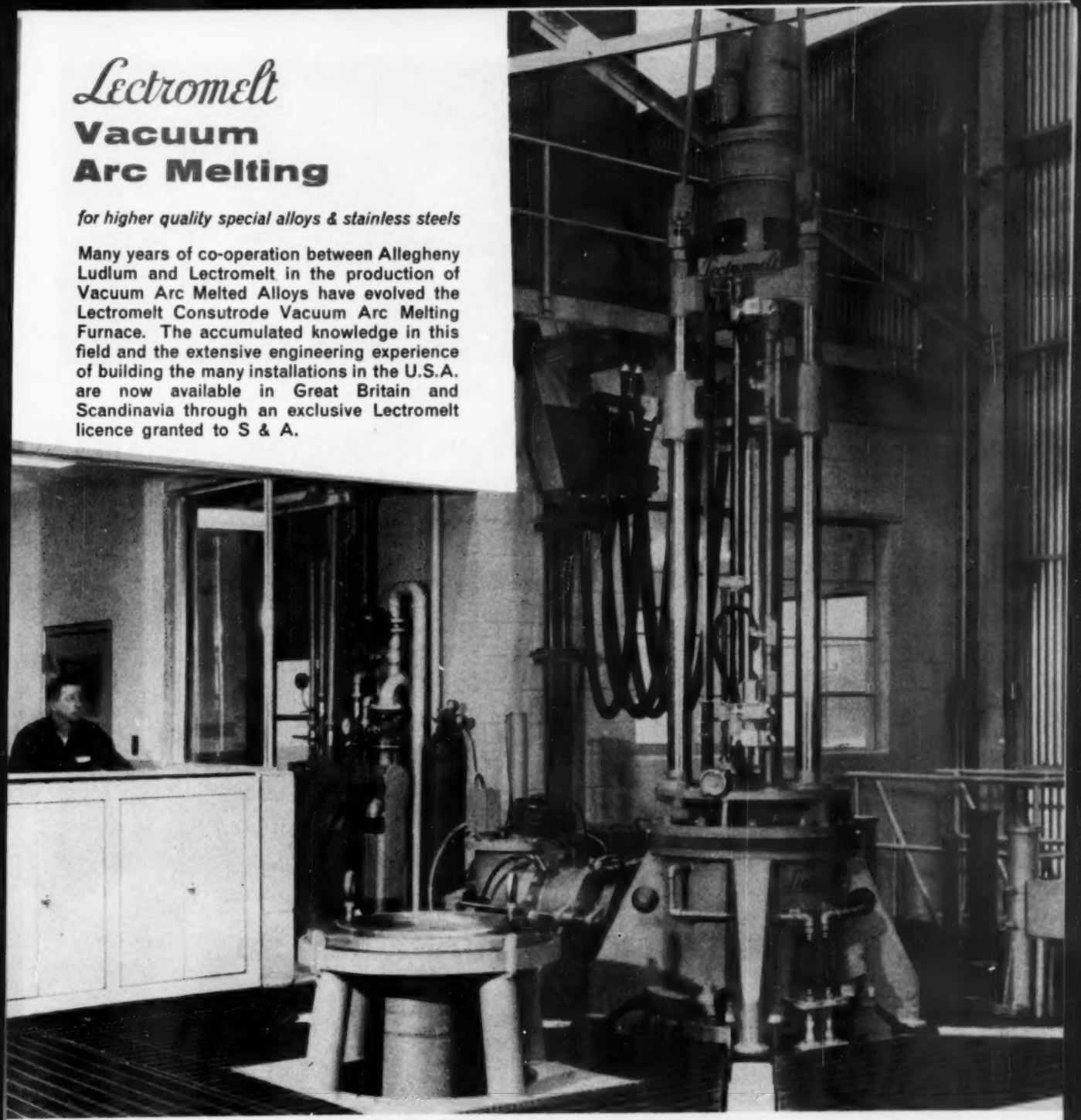

Lectromelt

A 20" x 8500 pound furnace installed by Lectromelt at Latrobe Steel Co., Latrobe, Pennsylvania.

Stein & Atkinson Ltd.

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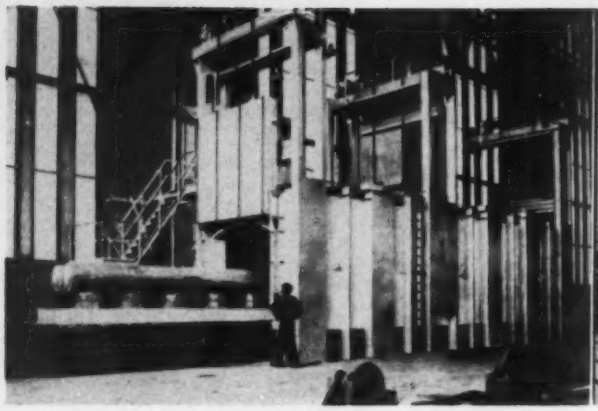
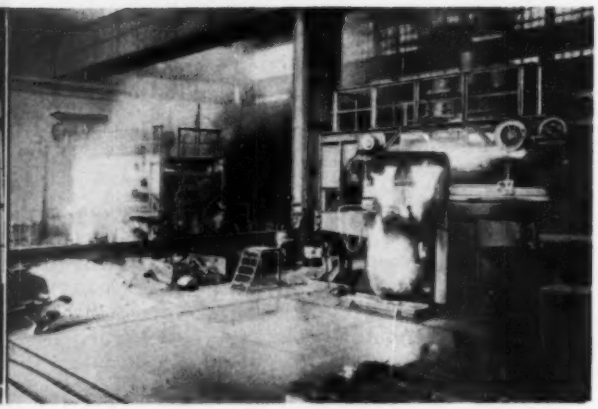
Broadway SA109



150 ton capacity bogie hearth furnace



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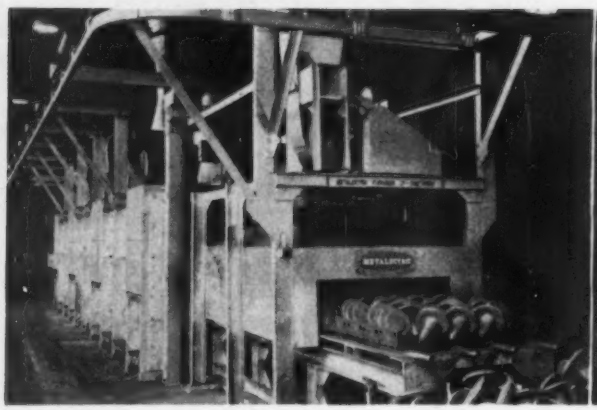

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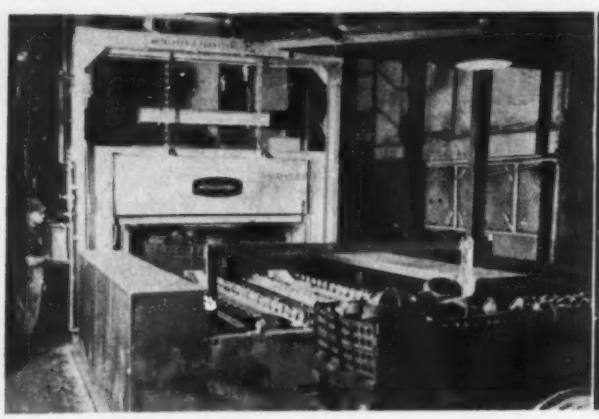
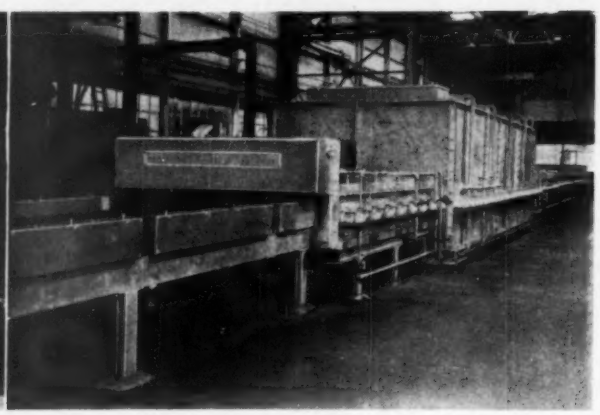


Continuous blackheart and pearlitic malleablizing



Roller hearth bright annealing

13/40D/60

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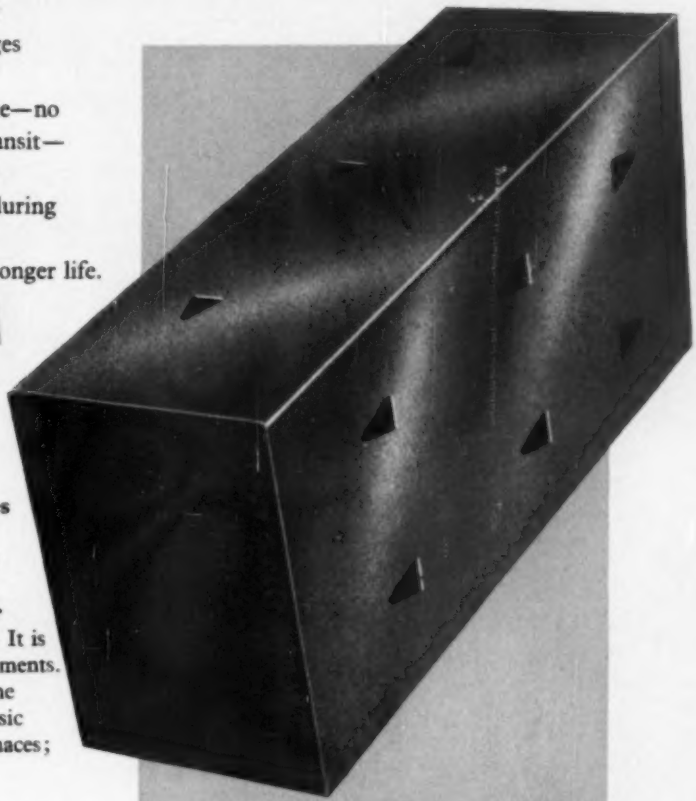
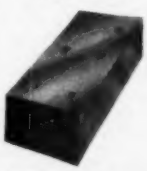
COVERED BY BRITISH PATENT Nos. 546, 220



For over 10 years General Refractories Ltd. have manufactured steel-clad basic bricks under licence by the Heuer processes. **One of the principal features of Ferroclad is that the graded refractory material and metal casing form an integral unit by means of controlled hydraulic pressure—no air infiltration—the case fits like a glove.** It is made in four main qualities to suit requirements. Ferroclad bricks are extensively used in the roof, front and back walls, and ends of basic open hearth furnaces; in basic electric furnaces; and in copper reverberatory furnaces.

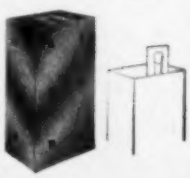
FERROCLAD 'EE' BRICKS



Divided internally by two plates placed lengthwise. This system reduces flaking and spalling to a minimum and can be applied to all Ferroclad shapes and sizes.

COVERED BY BRITISH PATENT Nos. 546, 220., 778, 047., and 778, 048

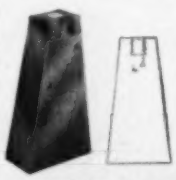
TABHANGER SUSPENSION



Bricks supplied with a special stainless steel tab co-moulded with the brick, embedded into the surface of the top face. By the use of a special tool the tab may be bent out at right angles to the top face to form a hanger.

COVERED BY BRITISH PATENT Nos. 546, 220 and 749, 491

FERROCLIP SUSPENSION



This method allows the brick to be suspended from the end. The Ferroclip hanger slot may be moulded into any roof or wall shape.

COVERED BY BRITISH PATENT Nos. 546, 220 and 600, 630

During recent years the demand for Ferroclad has increased so tremendously, that General Refractories Ltd. have granted a sub-licence to Messrs. Pickford Holland & Co. Ltd. to manufacture similar products within the scope of the British Patents mentioned above.

The basic process of metal-cased brick manufacture is covered by British Patent No. 546,220 and other additions, including the "Ferroclip," the "Tabhanger" and the "EE", are covered by British Patent Nos. 546,220, 600,630, 749,491, 778,047 and 778,048.

full technical information on request

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426

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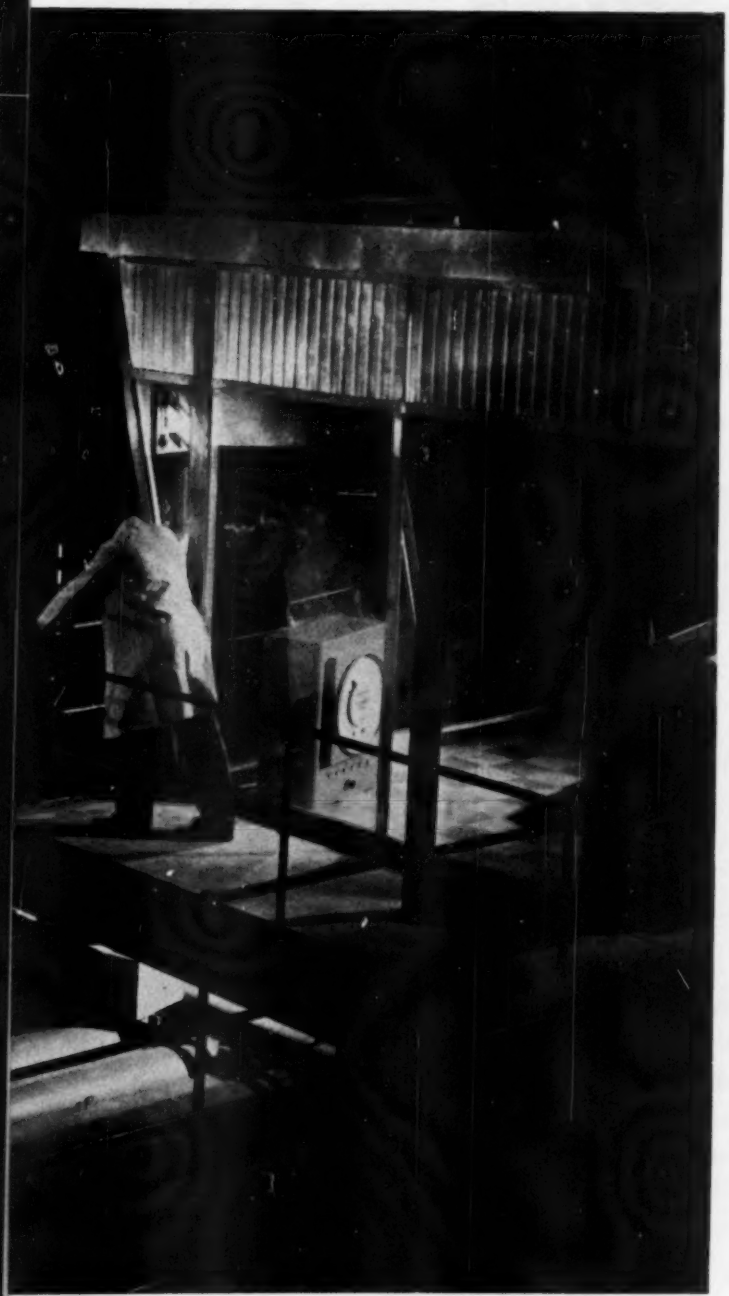
aluminium rolling mill in Europe



This 8,000 horse-power 144-inch mill, first stage in a 'hot line' one third of a mile long, is the centrepiece of the latest expansion of the continuous strip mill at Rogerstone, Monmouthshire. New equipment and modernisation at a cost of over £7 million makes Rogerstone the largest aluminium rolling mill in Europe, with a nominal capacity of 70,000 tons of sheet and strip products a year, plus a substantial extra capacity for hot-rolled coil and plate.
It demonstrates the determination of Alcan Industries – the new name of Northern Aluminium Company – to maintain their position as leading suppliers of aluminium products to British industry.
Aluminium alloy plate over eleven feet wide can be produced by this big new mill, compared with the maximum width previously available of about seven feet, and this wide plate offers the designer more scope and can save time and money in construction.
It is of special significance to the shipbuilding, railway, chemical, petroleum and other engineering industries.
This mill thus complements the other facilities at Rogerstone for production of high-quality, flat, accurately-sawn plate: a 4,000 ton stretcher and special plate saw.
Leadership in equipment is backed by long experience in the application of aluminium, including joining methods. If you use, or might well use, aluminium, for expert advice get in touch with your nearest Alcan Industries (formerly Northern Aluminium) office.

LIMITED

This new name identifies the company more clearly as a member of the Aluminium Limited of Canada enterprise, comprising some fifty companies in all parts of the world. Emphasis is thereby laid on the advantages that the company and its customers enjoy in being able to avail themselves of the Group's extensive research and development resources, and its great fund of experience in the smelting, manufacture and application of aluminium.

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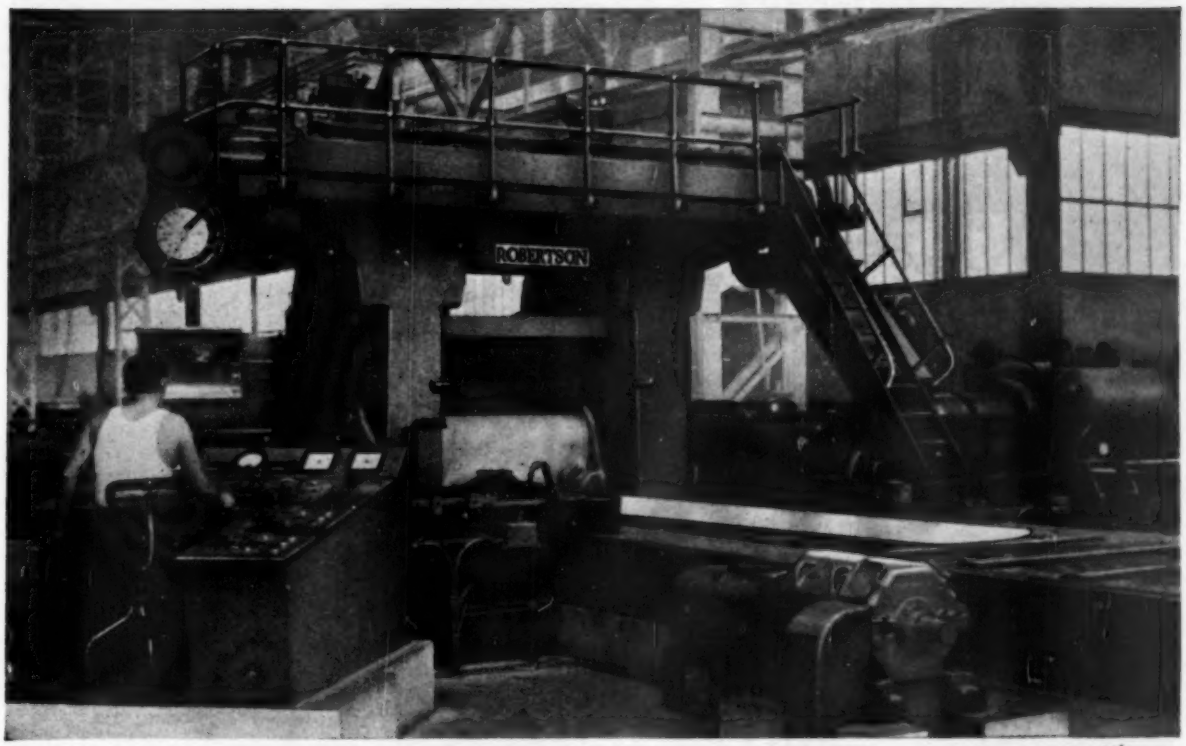

Photograph by courtesy of Compagnie Francaise des Métaux.

Two-high Reversing Hot Mill for aluminium installed by Robertsons at the works of Compagnie Française des Métaux.

Rolls 32" × 64". Speed 400 feet per minute.

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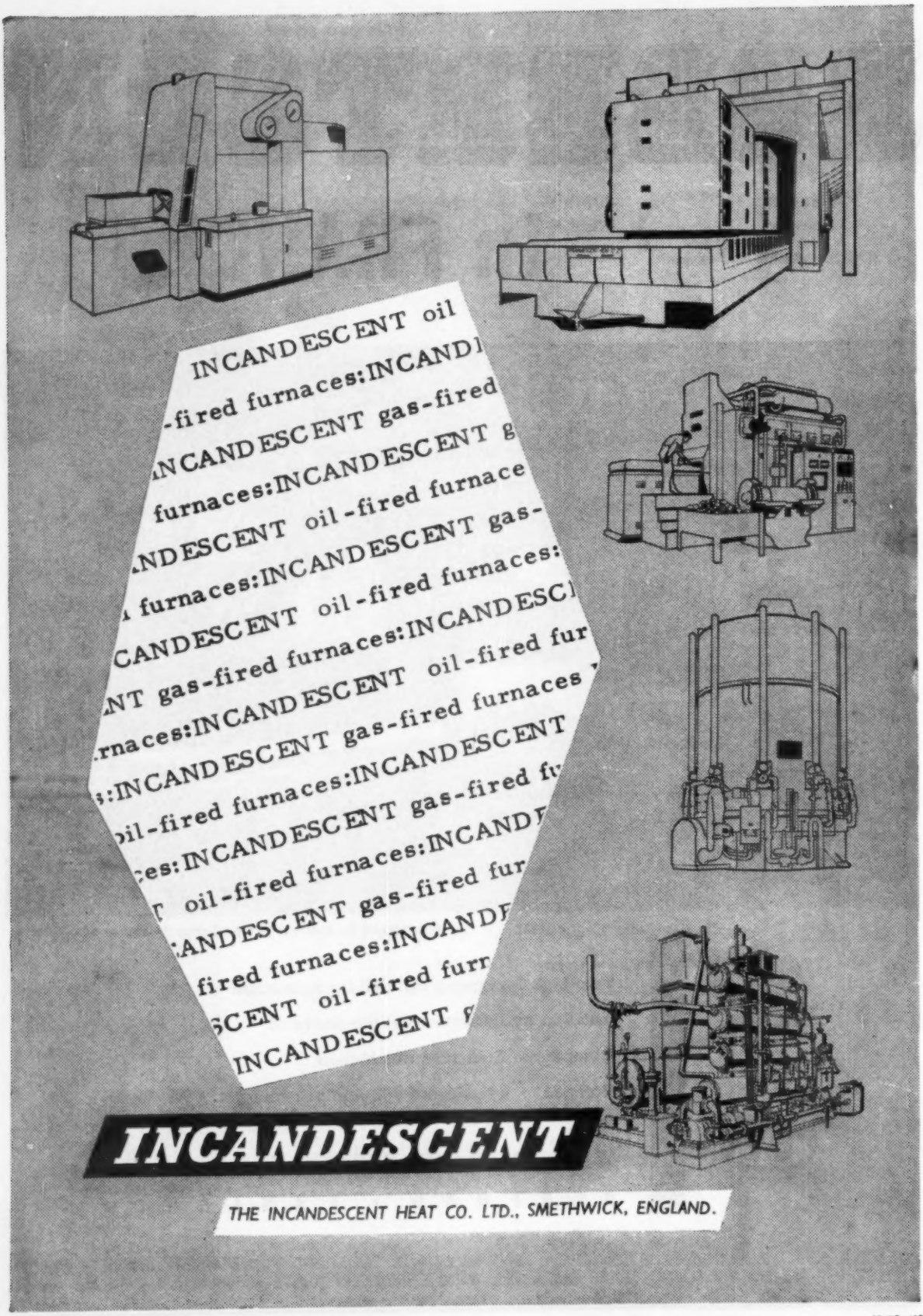
LICENSEES FOR THE BUILDING OF SENDZIMIR COLD REDUCTION MILLS AND PLANETARY HOT MILLS, HALLDEN GUILLOTINE AND ROTARY FLYING SHEAR MACHINES, AND TORRINGTON METAL WORKING MACHINERY. WT. 3388


INCANDESCENT oil
-fired furnaces:INCAND
INCANDESCENT gas-fired
furnaces:INCANDESCENT g
NDESCENT oil-fired furnace
furnaces:INCANDESCENT gas-
CANDESCENT oil-fired furnaces:
NT gas-fired furnaces:INCANDESC
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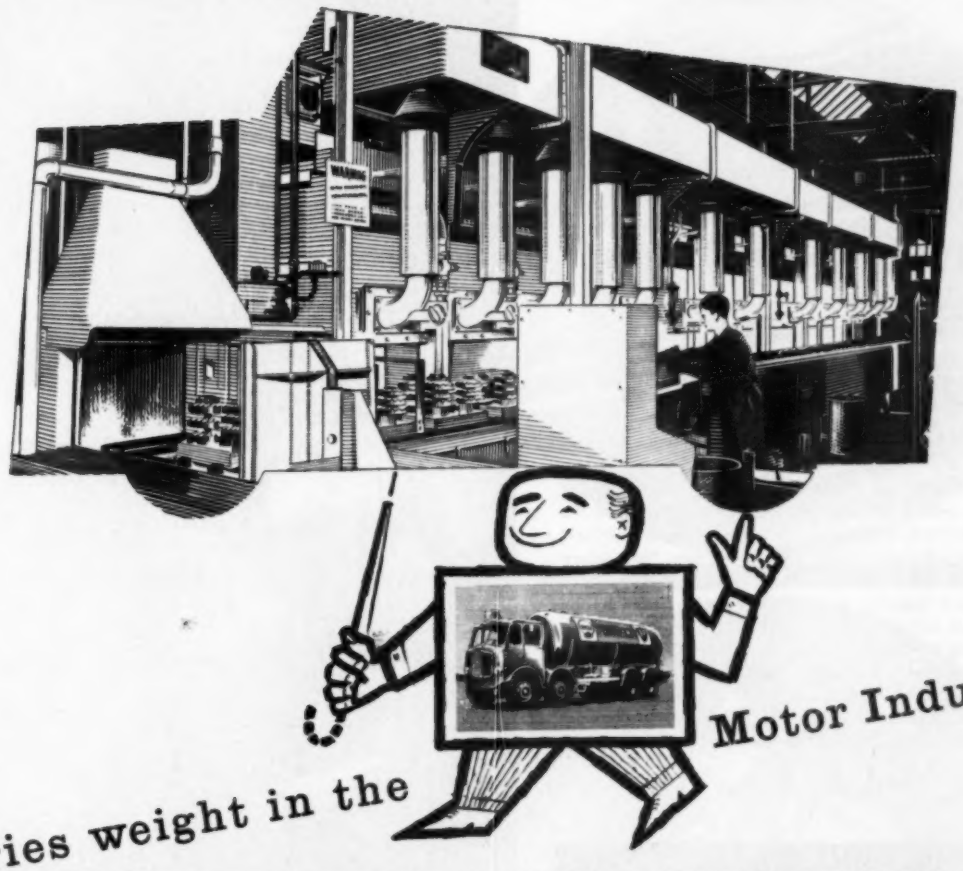
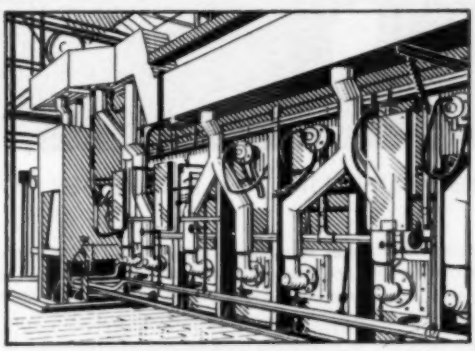
THE INCANDESCENT HEAT CO. LTD., SMETHWICK, ENGLAND.

PROPAGAS PROPANE



carries weight in the Motor Industry



These illustrations, by courtesy of Ford Motor Co. Ltd, show two of many continuous gas carburizing furnaces installed at their Dagenham factory, using endothermic atmospheres produced from PROPAGAS.

PROPAGAS provides industry not only with a high calorific value fuel gas (approximately 2,500 b.t.u. cubic foot) but also with an excellent medium for the production of special furnace atmospheres. It is widely used for gas carburizing, carbonitriding and bright annealing of ferrous and non-ferrous metals.

BOTTOGAS Butane, like Propagas, is a petroleum gas delivered and stored as a liquid under moderate pressure. Bottogas is used as a fuel for fork lift trucks and for many other specialised applications.

PROPAGAS Propane and **BOTTOGAS Butane** come from the great British refineries of the Shell, Eagle and BP Groups, backed by a technical service second to none and with a complete sales organisation covering the United Kingdom.

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*Registered Trade Mark.

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Jack Masters knows what he's talking about—and in almost all the world's major industries there are men who talk about us as Jack does. They'll tell you that CARBORUNDUM help them make better products, cut costs, and speed production. In the battle for expanding markets overseas, high quality, low price, and early delivery dates are vital for victory. There are rich prizes for those who can meet the challenge. Abrasive and refractory products by CARBORUNDUM can help *you* to win them.

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MATERIAL

RAY DETECTOR

Amount of rays passing through material indicates thickness or weight per unit area.

That's the very basic explanation of the Atomat principle. There are three main gauges; the Atomat Cold Strip Gauge, the Hot Strip Gauge and the Beta Gauge.
In addition there are special purpose variations, made to meet the requirements of many different industries.

HERE'S WHAT AN ATOMAT GAUGE CAN OFFER YOU

- **Time and material saving both in production and setting up**
- **High accuracy**
- **Calibration in your usual units of measurement**
- **Continuous readings for materials in constant production**
- **Auto standardisation and control if you require**
- **Minimum maintenance**

Baldwin Atomats are already boosting production and cutting costs in the paper, metal rolling, rubber and plastics industries. Highly successful applications also include the measuring of selenium on aluminium, coatings of paint and lacquers, density of wood chips, coatings of grit on abrasive papers and padding and resin impregnation in the textile industry. But the potential of Baldwin Atomat Nucleonic Gauges has hardly been tapped.
Is there a place for them in your plant?

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Send for the Baldwin Brochure MA/124. It gives full information on Atomat Gauges clearly and concisely.

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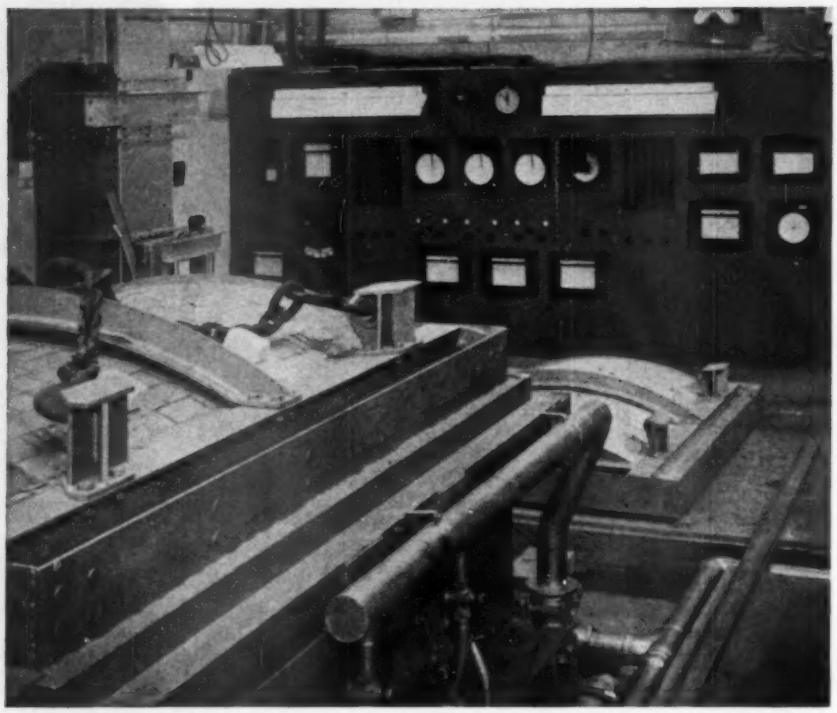
fully instrumented



B.I.S.R.A. begin a new chapter in research with their new experimental forge furnace. It will give the steel industry the answers to many long-standing questions on furnace design, operation and control

The Energy Section of B.I.S.R.A. is now studying how to improve efficiency in batch type heating processes. And Honeywell Instrumentation is helping to write up the essential facts and figures —programming the heating cycles, too.

MULTI-RECORDING. Four ElectroniK 12-point Recorder/Controllers continuously record the data from more than 100 thermocouples dispersed in the walls, chamber, flues, air ducts, recuperator, stack of the new experimental forge furnace, and in the 3-ton ingot.

PROGRAMME CONTROLLING. A master temperature Controller sets the heating cycle and governs secondary controllers for fuel flow. At the same time, other Honeywell instruments keep a critical eye on furnace pressure, or help the analysis of flue gases.

BIOGRAPHICAL NOTE. Similar instruments are used throughout B.I.S.R.A's laboratories. Versatile, accurate, they are adaptable to pure research, pilot plant operation and to process control on the largest scale. In every application they are made doubly valuable by the design, engineering and maintenance service they carry with them from Honeywell Head Office or from branches in the principal towns and cities in the United Kingdom.

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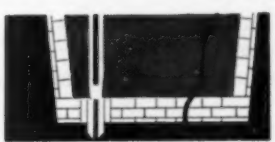

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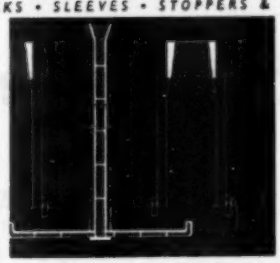

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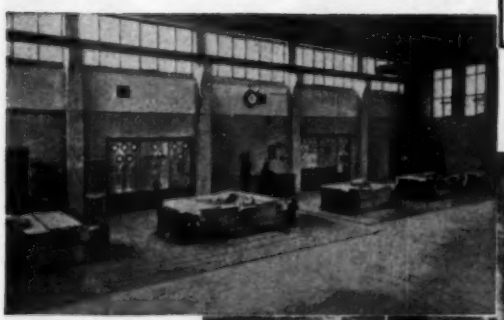
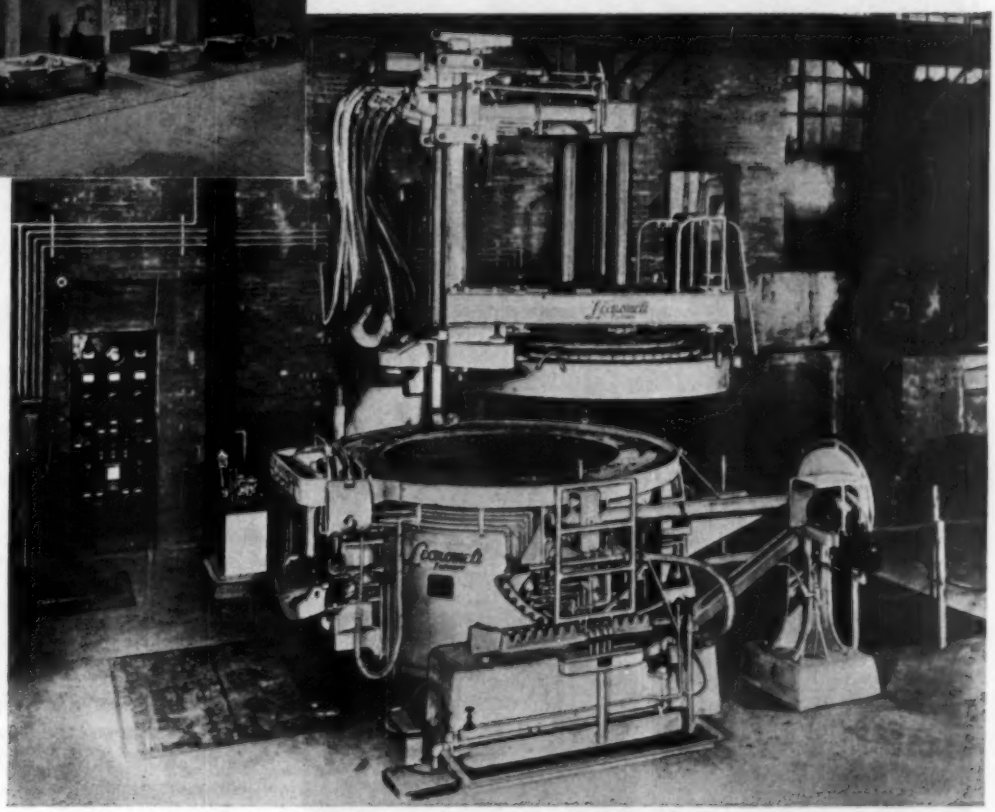
GWB

DEMAG Lectromelt





Demag high-frequency induction furnace installation at a steel making plant, incorporating two 1 ton capacity furnaces, two 660 lb. capacity furnaces, and one 110 lb. capacity furnace.

A 3 ton capacity Lectromelt arc furnace of 9' 0" diameter shown with roof swung ready for charging.

High Speed and High Output are the key features in the medium and smaller range of furnaces offered by G.W.B. Furnaces Ltd. Many years are behind both the Coreless and Arc Furnaces of Demag and Lectromelt designs.

Daily, electric furnaces are finding wider and wider applications, and the coreless induction (crucible) type is no exception as it is an ideal medium for melting steel and iron, and all other metals. It is particularly advantageous in steel works where a charge of scrap of known analysis is melted and is an installation that pays for itself in a very short time. The proven economies of operation of electric arc furnaces offered by G.W.B. have earned an enviable reputation, and a few of their outstanding features are:—

Separately mounted roof lift and swing mechanism for top charging; Four point roof suspension of the roof ring; Electrode holders of the power operated, spring-held, air release type; High speed movement of the electrodes; Offset rocker centres to return furnace from extreme tilt in event of power failure.

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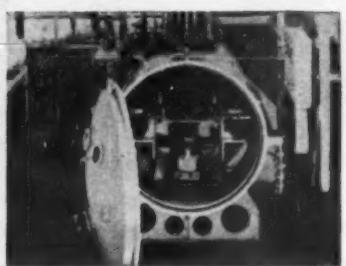
Model 2555 Vacuum Induction Furnace with melting capacity of 50 pounds of steel. Other standard furnaces have capacities of 12 to 3,000 pounds.



Model 2705 Non-Consumable Arc Skull Furnace with a capacity of 50 pounds of titanium. Other standard vacuum arc furnaces have capacities of 8 to 10,000 pounds of titanium.

Metals for parts to withstand jet engine temperatures and stresses can be produced in vacuum furnaces. Such metals enable components to be made with fewer rejects. If your products need parts that operate under severe conditions or need high purity metals or alloys—a vacuum furnace will help you to produce them.

Wild-Barfield can supply equipment made to the designs of and tested by the unrivalled experience of the National Research Corporation who have built and operated more high vacuum furnaces than any other company in the world.

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WB 84

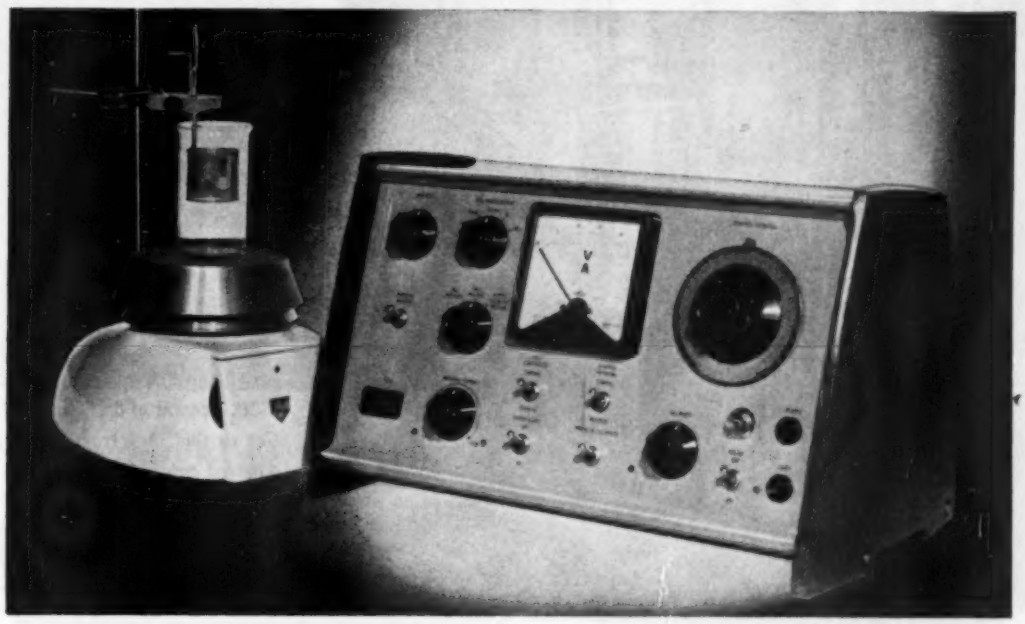
A 1654

Wadsworth Controlled Potential Electro-Depositor

ELECTRO-DEPOSITION has long been recognised as an analytical and separative technique for solutions containing metallic ions. The method may be applied where the ionic concentrations of the metals present in solution vary widely, separation being best achieved where the potential of the working electrode relative to the solution is controlled, using a reference electrode as described independently by Sand & Fischer. Manual control of the electrode potential, although possible, is tedious and time consuming, with the result that automatic control circuits have been developed by a number of workers.

The Southern Analytical controlled potential electro-depositor is based upon a design due to N. J. Wadsworth of R.A.E., Farnborough, and is intended primarily for use with platinum electrodes. The circuit is fully transistorised in the interests of compactness and reliability and is capable of supplying a maximum controlled current of 10 amperes to the electrolysis cell. In operation, the required cathode potential is preset upon a built-in high grade potentiometer. Any error between the preset potential and that of a reference half-cell (e.g. saturated calomel), mounted immediately adjacent to the cathode, is applied to an amplifier which controls the current passing through the cell in such a sense that the potential error tends towards zero.

In order to ensure that thermal drift in the control amplifier is held at a very low level, a water-cooled heat sink is employed to maintain the transistors at a substantially constant temperature. The accuracy of control attained may be judged from the fact that the incremental error in electrode potential as a function of cell current has a mean value of 1.6 millivolts/ampere; thus, when the electrolysis current has fallen to the low levels obtaining towards the end of a deposition, extremely close control is established. It is noteworthy that the potential defined by the built-in potentiometer cannot be exceeded as cell current cuts off at zero potential error.

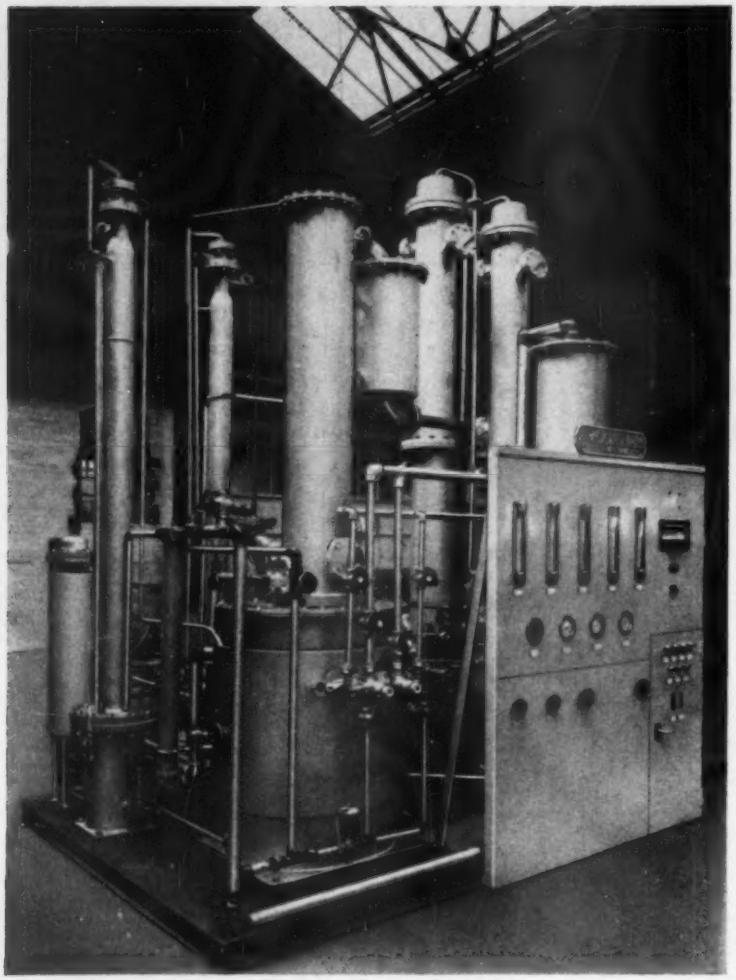


The apparatus is supplied complete with leads for connection to the user's own electrode assembly. If desired however, suitable standard accessory equipment can be provided.

SOUTHERN *Analytical*

SOUTHERN ANALYTICAL LIMITED CAMBERLEY SURREY TELEPHONE: CAMBERLEY 3401

GM v L MONO I GENERATORS



. . . . are Exothermic

. . . . are primarily designed for application where a high CO/CO_2 ratio is necessary such as gas carburising, carbo-nitriding, clean hardening, sintering etc.

. . . . can be used for the production of inert gas containing only traces of CO_2

. . . . can be fired by any fuel gas or a light fuel oil.

The MONO I Generator illustrated produces 1,000 c.ft. per hour of a gas having a Dewpoint of—15°C. and a CO_2 content of 0.2% using Town's Gas as fuel. Any trace of oxygen is removed catalytically.

G.A.A. ATMOSPHERE GENERATORS

. . . . simple to operate

. . . . consistent in performance

. . . . fully protected by automatic safety devices

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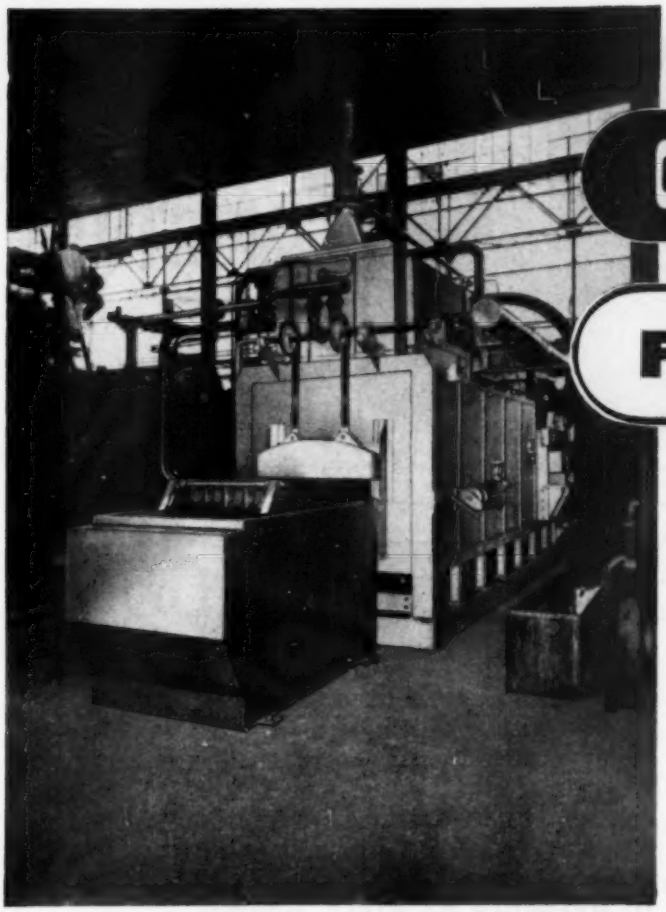

GIBBONS

Patent Nos. 637,337. 646,691. 606,401.

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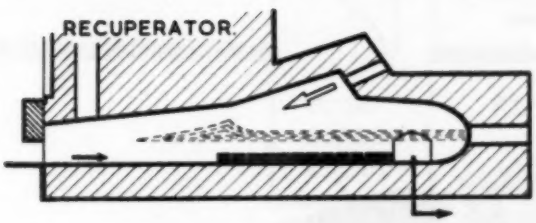
No Scale

Longer Tool Life

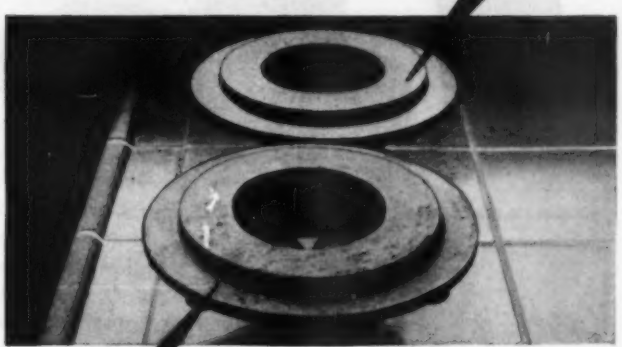
Low Fuel Cost

No Metal Loss



PRO-TEK Continuous Billet Heating Furnaces are built under licence arrangements with Metallurgical Oxygen Processes Limited and Indugas, G.m.b.H., Essen.
Specially suitable for repetition work either mild steel or alloys.
Standard sizes from 10 cwts. per hour upwards with guaranteed performance and scale free high quality finish heating.

PRO-TEK FORGING



NORMAL FURNACE FORGING

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G105

Concentration

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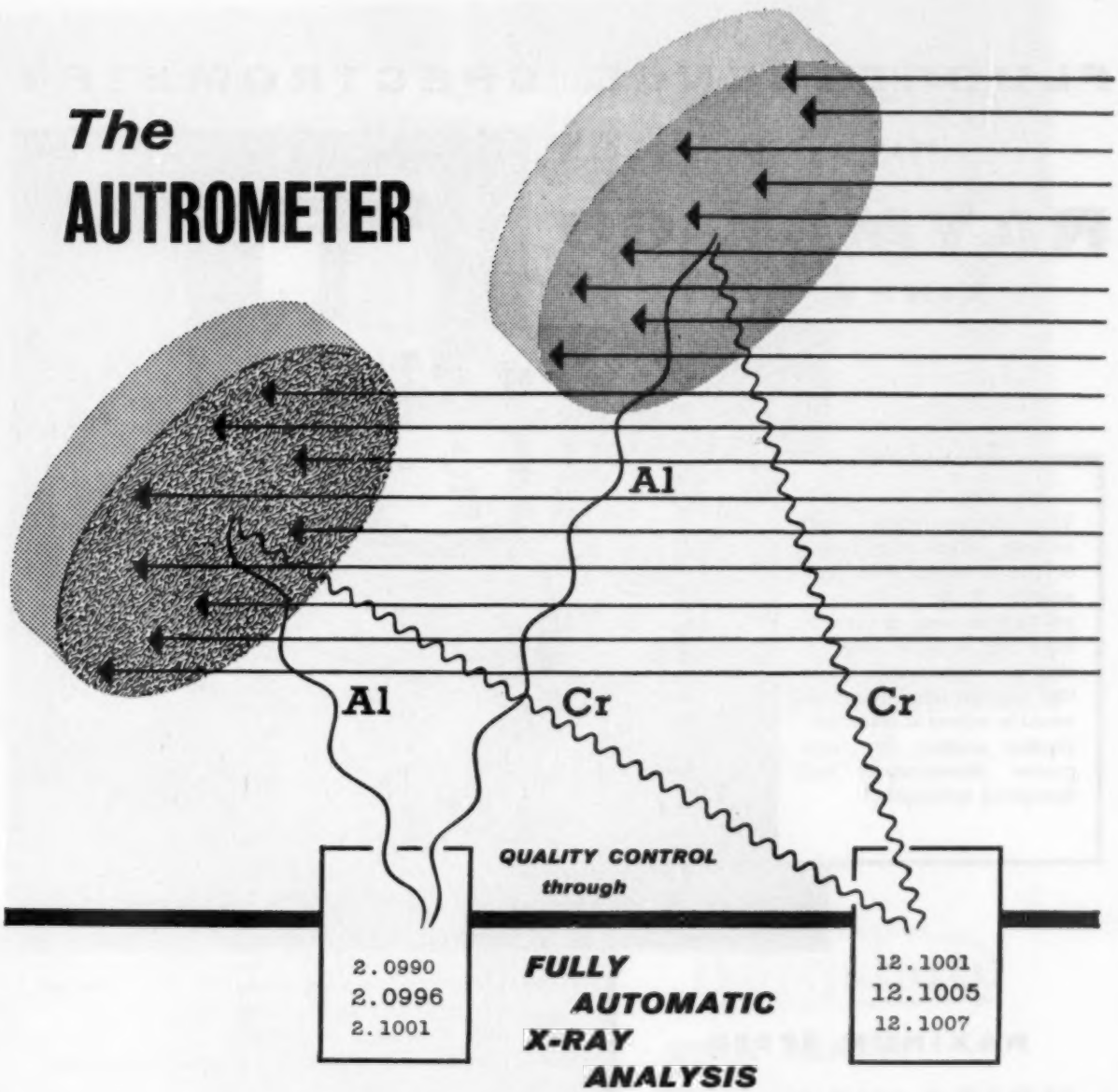

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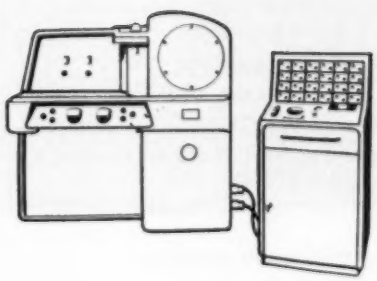

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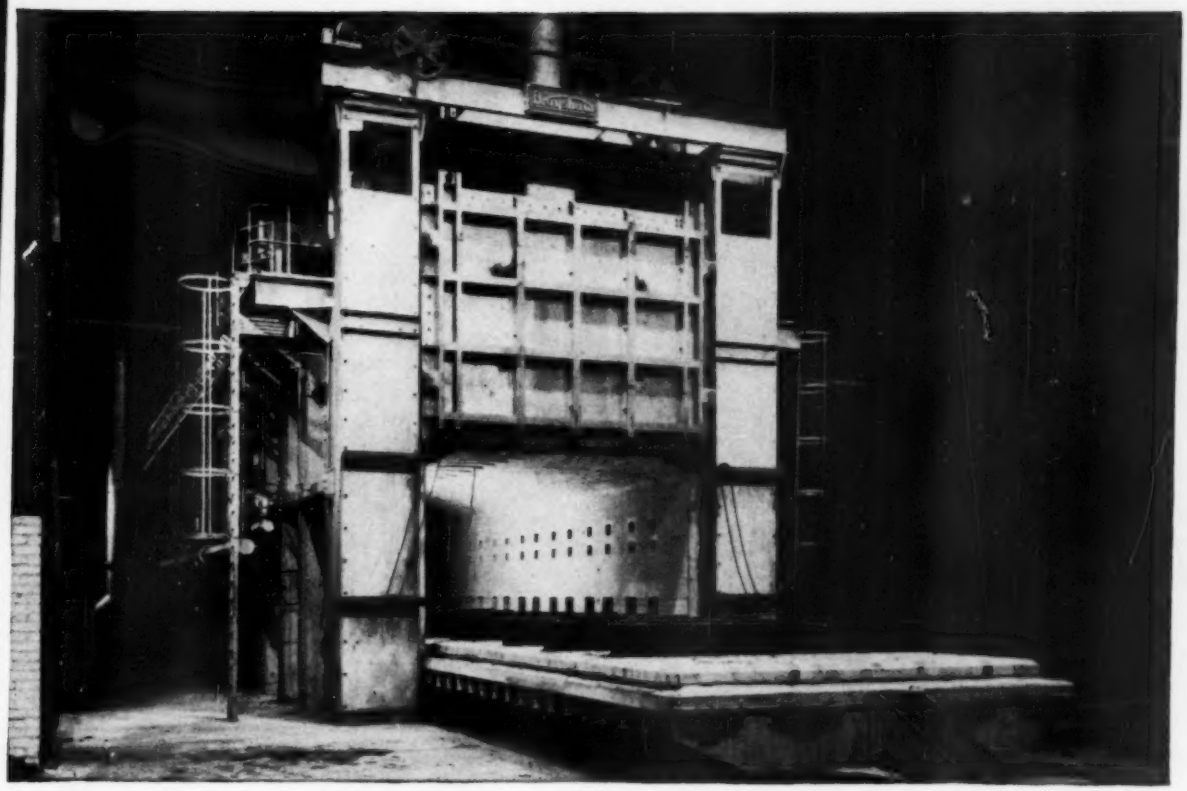

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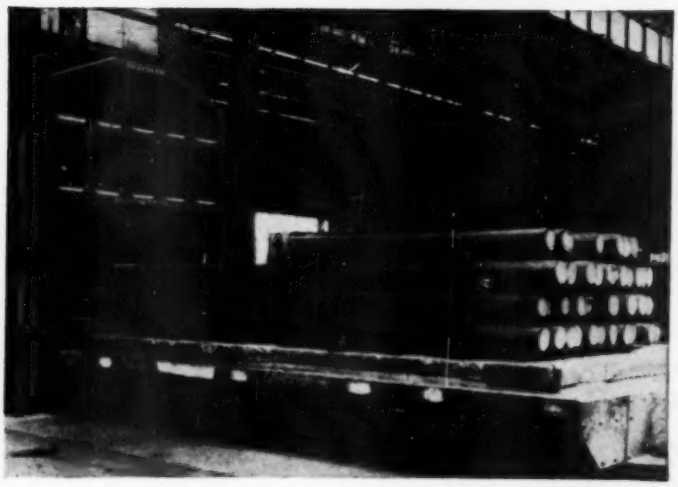

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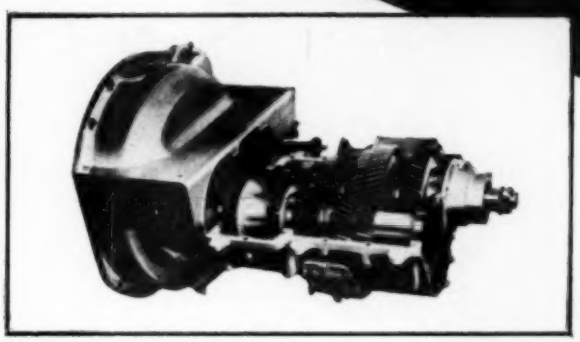

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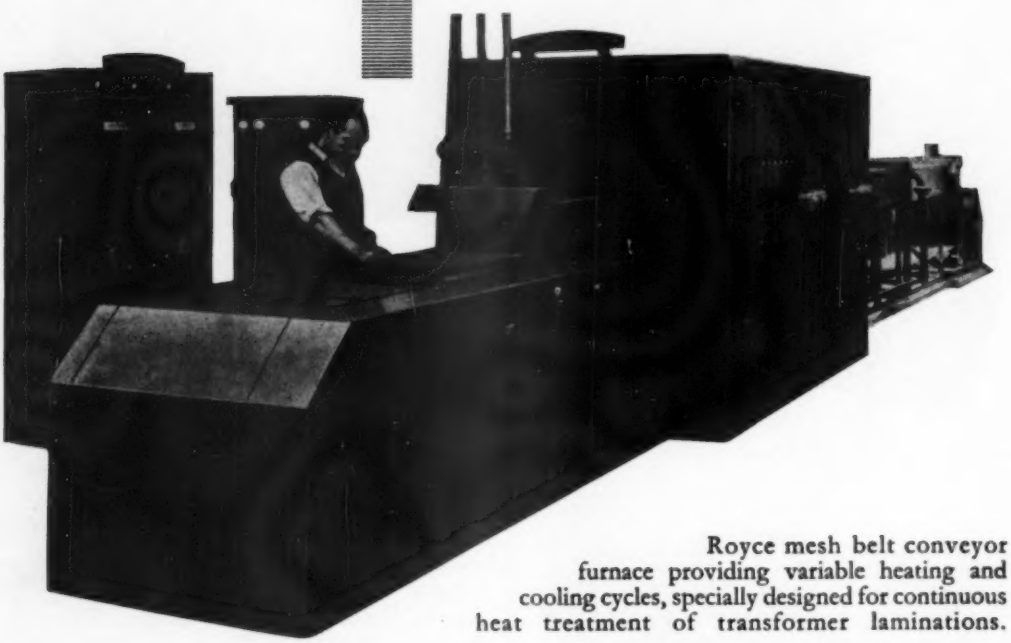

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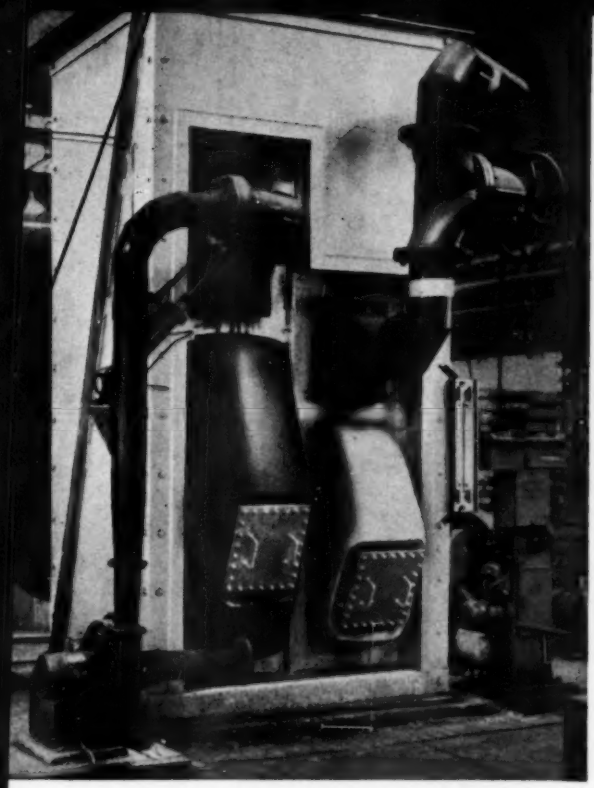

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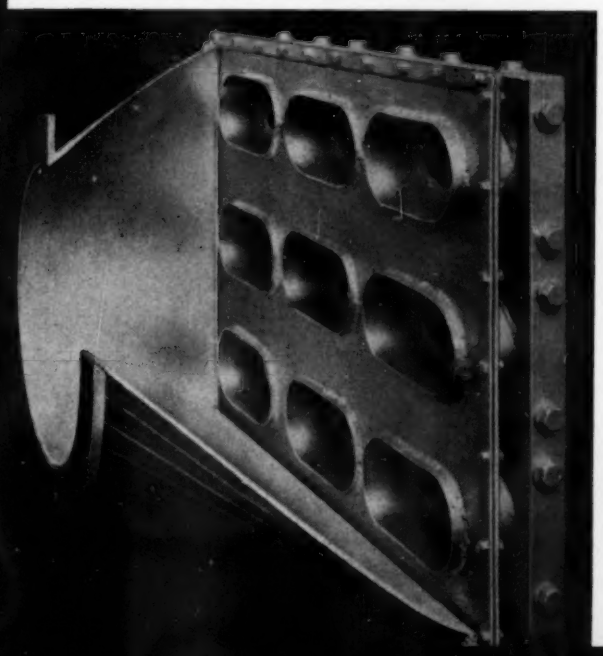

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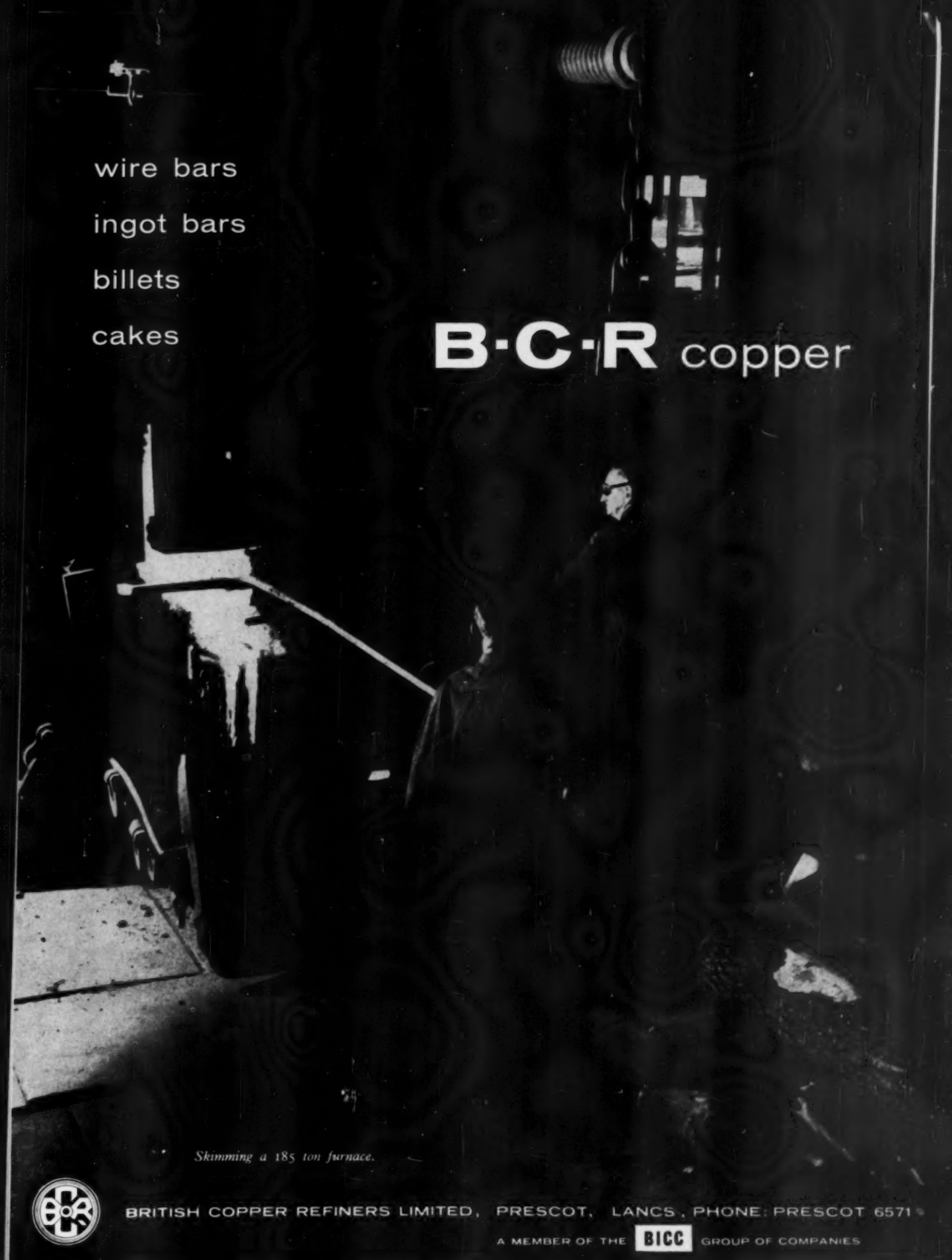

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THE BRITISH JOURNAL OF METALS
INCORPORATING THE METALLURGICAL ENGINEER

CONTENTS FOR NOVEMBER. 1960 Vol. 62 No. 373

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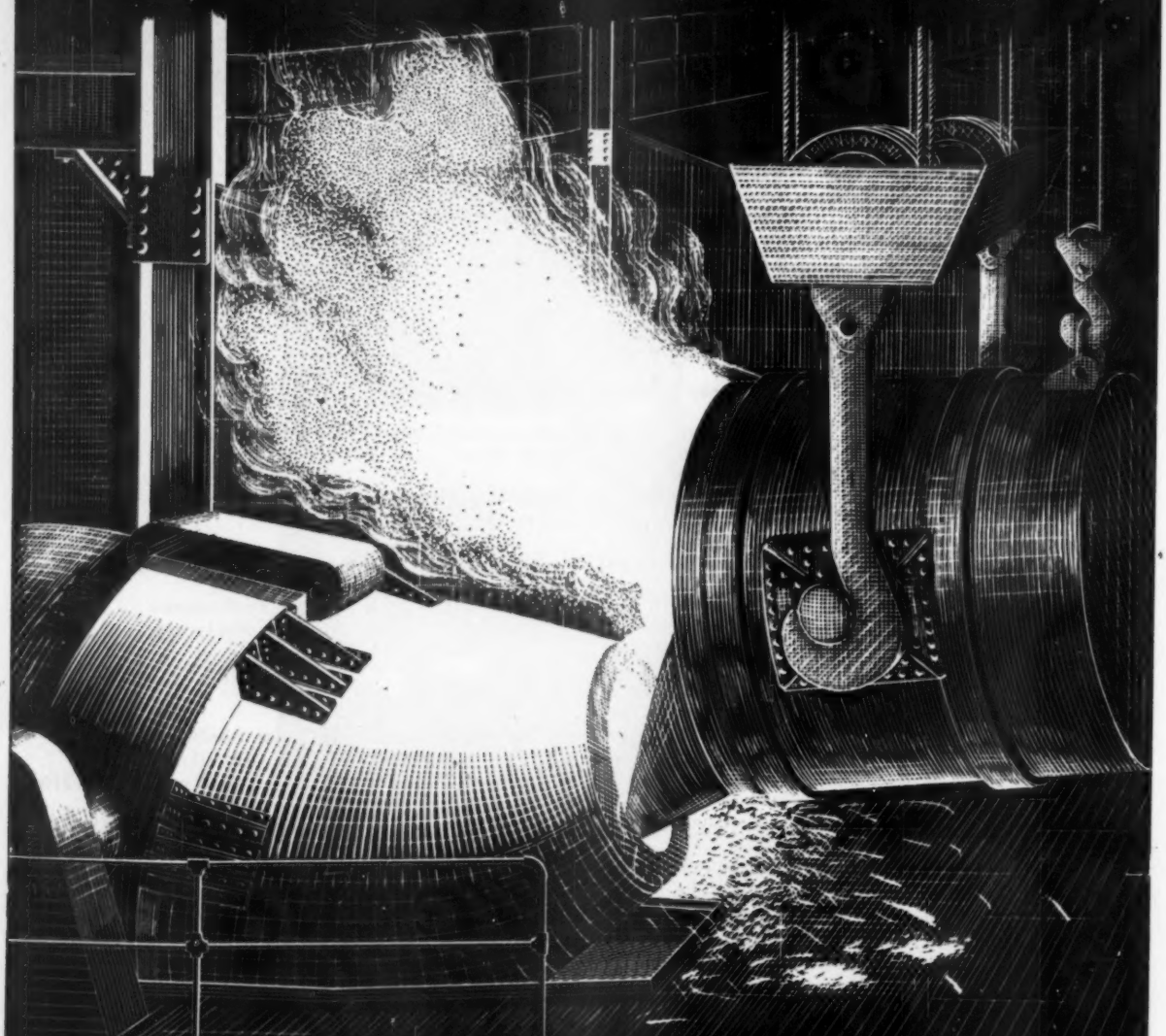

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METALLURGIA

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NOVEMBER, 1960 Vol. LXII. No. 373

Welding in Russia

IF ever there was a time when anyone doubted the extent of the technological progress made by the U.S.S.R., that time is long past, for the doubts have been effectively dispelled by the demonstrations of such progress provided by the various "sputniks" that have been put into orbit in the last few years. While spectacular developments such as these capture the popular imagination, they represent a great scientific achievement, and are, moreover, based on an advanced state of technical development in many supporting fields. Welding is one such field, and the impressions gained by a party of experts from the United Kingdom in the course of a two weeks' visit to welding research institutes in the Soviet Union are of special interest.

There has been a tendency for the scepticism of the man in the street to have been swept away by the recent Soviet scientific "show of strength" and for there to have been engendered a belief in the technological infallibility of the Russians. The scientifically trained mind would not be so influenced, of course, but would appreciate that, despite the disproportionately large numbers of Russian scientists and technologists, the considerable amount of effective work being carried out in the laboratories and workshops of the United Kingdom is making a not inconsiderable contribution to the advance of scientific knowledge. It is not surprising, therefore, to learn that the welding mission found that in some spheres the U.K. lags behind the U.S.S.R., whilst in others the reverse is the case. Members of the delegation returned with the impression that emphasis there is placed more on the application of new welding processes, and the associated problems, than on fundamental work for the sake of increasing knowledge. This arises from the all-out drive to expand production and the clear realisation of the part played by welding toward that end. The importance of welding in the modern engineering and constructional programmes is probably more deeply appreciated in Russia than in this country.

The visit took place under the auspices of the U.S.S.R. State Scientific and Technical Committee, and was arranged to coincide with the opening of a welding exhibition in Moscow within the permanent Exhibition of the Economic Achievements of the National Economy in the U.S.S.R. The party was impressed by the standard set by this exhibition and by some of the exhibits, particularly in the fields of cold pressure welding (dissimilar metals), friction welding and diffusion welding. Automation and mechanisation of welding processes was the dominant theme. Specific items of interest were a portable flash welder with contour transformer to deal with pipes up to 27 in. in diameter; an automatic twin head circumferential CO_2 welding machine for the same size of pipe; and a whole range of tungsten arc welders dealing with pipes in a variety of materials and sizes from $\frac{3}{8}$ in. to 7 in. diameter.

Methods to overcome the difficulties of distance and transportation to outlying areas were in evidence, and storage tanks up to 50 ft. diameter were shown which are prefabricated and rolled up for transport to site where the rolls are subsequently pulled open by means of tractors and assembled with the minimum of site welding. Pipelines are dealt with in the same way, being welded in the flat and coiled for despatch. Using a tractor-pneumatic combination, workers expand the 600 yd. coils into cylinders on site. The acceptance standard for the finish of welded components is lower than in this country; puckering on the face of sheet materials when welding on studs, for example, is accepted in the manufacture of car bodies. This, however, is considered of secondary importance to the functional qualities of what is produced.

Although the exhibition was clearly intended to accelerate the spread of the latest techniques into the industries of the Soviet Union, it was significant that even quite senior staff of some of the institutes visited by the British party had not been to the exhibition, nor was it likely that they would be able to go. This contrasted sharply with the sight of whole families—including small children—visiting the exhibition on Sundays.

Of the five research institutes visited only one was entirely devoted to welding. One of the others was the research laboratory of a factory devoted to the manufacture of arc and resistance welding equipment; another was equivalent to a university department; and in two others welding was a department in an institute which had very much wider terms of reference. In only one of the institutes—the Baikov Institute in Moscow—is most of the research fundamental. In the other institutes, although some fundamental work is carried out, most of the research is directed towards the eventual development of new welding processes and new processes of manufacture.

Whilst the research and development effort in the Soviet Union in welding technology is on a very much bigger scale than it is in this country, this is to be expected from the difference in size between the two countries. There are some quite outstanding achievements, but there are also certain spheres where, owing to the special conditions in the Soviet Union, work is not as far advanced as in this country. Welding of aluminium alloys by the inert gas metal arc welding process appears to be further advanced in this country than in the Soviet Union. On the other hand the emphasis on submerged arc welding has led to the development of methods and suitable fluxes for the welding of aluminium alloys, titanium alloys, and copper. The joints appear to be of good quality and are claimed to have properties not very inferior to those of the parent material.

The conditions prevailing in many parts of the Soviet Union are unique, and processes may be developed under the pressure of economic factors different from those

existing outside that country. Friction welding, for instance, can sometimes serve as an alternative to resistance flash welding and requires less power. The urge to develop this process came from the possibility of its use in areas of very limited power supply. Another illustration based on difficulties of supply is bare wire welding using steam as the shielding gas, instead of argon or carbon dioxide.

Very little has been done in the U.K. to date with the friction welding and diffusion welding processes to which Russia is now paying so much attention. These processes appear to offer a number of advantages, however, and the British Welding Research Association is to study their potential applications. In the friction welding process, which employs no external heat, the components to be welded are mounted in a device similar to a lathe, having a rotating headstock and stationary tailstock. The rotating and fixed components are brought into contact by pneumatic or hydraulic pressure, when intense local heat is generated. At the moment of upsetting pressure reaches maximum and the drive is automatically stopped. The process can obviously only be used where at least one of the components is a cylindrical or disc-shaped section, but it is claimed that the conversion of electrical energy into motive force is more economic than conversion into heat through a transformer, due to the inductance losses in the transformer secondary circuit ; further, the process lends itself readily to mechanisation. The diffusion welding process consists, in essence, of bringing the clean surfaces of the components together in a vacuum when the application of heat and pressure results in the atoms diffusing at the facing surfaces to produce an alloy. Joints in dissimilar materials, e.g. metal to non-metal, can be produced in this way.

One of the features of the Soviet system that impressed the visitors was the shortness of the time interval between the development of a new process or method and the availability of equipment for industry. This is probably due to the research workers having excellent facilities at their disposal for the manufacture of prototypes and production machines.

The party found the Soviet scientists, with whom they came into contact, extremely well informed on the literature of the West, but they greatly welcomed the opportunity of being able to ask questions on points which were not clear to them. They were very keen to receive considered critical comments on their papers, and expressed the desire to publish such criticism in their own journals.

Meeting Diary

21st November

Institute of British Foundrymen, London Branch (East Anglian Section). " Shaw Process," by NOEL SHAW. Lecture Hall, Public Library, Ipswich. 7.30 p.m.

22nd November

Institute of Metal Finishing, South West Branch. " American Developments in Plating Zinc Alloy Diecastings." Chairman's address. Royal Hotel, Cardiff. 7.30 p.m.

Sheffield Metallurgical Association. " Some Aspects of Research for Steel Foundries," by DR. A. H. SULLY. **Joint Meeting with the Sheffield Society of Engineers and Metallurgists.** BISRA Laboratories, Hoyle Street, Sheffield, 3. 7 p.m.

23rd November

Manchester Metallurgical Society. " New Aspects of the Electron Theory of Metals," by PROF. G. V. RAYNOR. Headquarters of the Manchester Literary and Philosophical Society, 36, George Street, Manchester. 6.30 p.m.

24th November

Institution of Engineering Inspection, North Western Branch. " Impregnation of Castings," by A. YOUNG. Engineers' Club, Albert Square, Manchester. 7.30 p.m.

Institute of Metals, Birmingham Local Section. " General Metallurgical Problems in the Motor Industry," by R. J. BROWN. College of Technology, Gosta Green, Birmingham. 6.30 p.m.

25th November

Institute of Metal Finishing, Sheffield and North East Branch. " Architectural Colour Anodising," by A. W. BRACE. Grand Hotel, Sheffield. 7 p.m.

29th November

Institute of Metals, Oxford Local Section. Junior Members' Meeting. " Electron Bombardment." (a) " Metallurgical Applications of Electron Probe Micro Analysis," by V. D. SCOTT ; (b) " Electron Beam Melting," D. B. GASSON ; (c) " Theory of Electron Probe Micro Analysis," by N. SWINDELLS. Cadena Cafe, Cornmarket Street, Oxford. 7.15 p.m.

Sheffield Metallurgical Association. " Some Applications of Castables and Mouldables in the Iron and Steel Industry," by DR. R. W. GRIMSHAW. BISRA Laboratories, Hoyle Street, Sheffield, 3. 7.30 p.m.

30th November

Institute of British Foundrymen, London Branch (Southampton Section). " Running and Gating of Castings," by W. W. GLICK. Technical College, St. Mary's Street, Southampton. 7.30 p.m.

1st December

Institute of British Foundrymen, London Branch. Joint meeting with the Institute of Metals, London Section. " Origin, Detection and Elimination of Gases in Cast Metals," by D. V. ATTERTON. 17, Belgrave Square, London, S.W.1. 6.30 p.m.

Liverpool Metallurgical Society. " Low Temperature Brazing," by H. R. BROOKER. Department of Metallurgy, The University, Liverpool. 7 p.m.

Society of Chemical Industry, Corrosion Group. " The Influence of Water Movement on Corrosion." (a) Ferrous Materials ; (b) Non-ferrous Metals, by (a) G. BUTLER and (b) N. V. NOWLAN. Olympia Empire Restaurant, London. (Tickets issued to members.) 2 p.m. and 3.45 p.m.

5th December

Institute of British Foundrymen, Sheffield Branch. " Engineering the Re-Layout of a Jobbing Core Shop," by A. ATHERTON. Technical College, Pond Street, Sheffield. 7 p.m.

6th December

Institute of Metals, South Wales Section. " ' Valence ' in Metals and Alloys," by G. V. RAYNOR. Metallurgy Department, University College, Singleton Park, Swansea. 6.30 p.m.

Sheffield Metallurgical Association, Analysis Group. " Atomic Absorption," by R. LOCKYER, BISRA Laboratories, Hoyle Street, Sheffield, 3. 7 p.m.

7th December

Institution of Plant Engineers, Southern Branch. " Treatment of Industrial Effluents," Polygon Hotel, Southampton. 7.30 p.m.

Institute of Welding, Manchester and District Branch. " Automatic Welding," by J. A. LUCEY. Reynolds Hall, Manchester College of Science and Technology. 7.15 p.m.

Manchester Metallurgical Society. " The Relationship between Physical Test Results and Engineering Performance," by H. J. LAMBLE. The Manchester Literary and Philosophical Society, George Street, Manchester. 6.30 p.m.

Society of Chemical Industry, Corrosion Group (Midlands Branch Meeting). " Cavitation," by D. J. GODFREY. Engineering Centre, Stevenson Place, Birmingham. 7 p.m.

(*continued on page* 210)

High Temperature Carburising

By T. W. Ruffle, F.I.M.

Ipsen Industries, Inc.

In view of the growing interest in gas carburising at temperatures higher than are normally used, the author discusses the type of equipment required and the means of controlling the carburising atmosphere, and concludes with the presentation of the results on a series of test runs.

DISCUSSION at a recent conference on carburising, organised by the British Iron and Steel Research Association and held at Harrogate, showed there is still widespread interest in the new development of carburising at higher temperatures. Some comments on current developments in this field and a more detailed account of work on atmosphere control at Ipsenlab, Rockford, U.S.A., may therefore be of interest.

Development work reported by the steelmakers at this conference certainly gave encouragement on the score of core properties after treatment at quite high temperatures, and some favourable indications on case structure were noted as well. In the U.S.A. reports are now becoming available indicating substantial progress in regular commercial application of temperatures up to 1,035° C., with some work indeed being carried out at 1,100° C. This is particularly the case with certain West Coast operators dealing with oilfield equipment, which is subject to severe duty and normally calls for heavy case-depths. In addition to substantial gains in economy of operation, favourable structures and properties appear to be developed in some instances by high temperature carburising as compared with carburising at more normal temperatures.

Distortion has been studied with particular care in one instance—the bore of a bushing in which normally the only operation after case hardening is honing. High temperature operation has proved quite well able in this case to maintain consistency and to give close control of dimensions. The honing operation (limited to maximum shrinkage of 0·0007 in. in a 1 in. bore) can easily be retained in place of the alternate expensive grinding operation after carburising at temperatures of up to 1,000° C.

Development work in Great Britain is proceeding in many places, with encouraging results in some at least, but no details have been generally released as yet. This particular process requires evaluation with some care, and experimental work is often handicapped by lack of suitable equipment, as furnaces capable of operating regularly at such temperatures have only recently become available.

Suitable Equipment

Suitable equipment is a most critical factor in this development for two reasons. First, furnaces of conventional construction cannot be pushed much above the usual operating temperature of 925° C. without excessive maintenance costs arising. Historically, it may be an interesting point as to whether the conventional carburising temperature was based on metallurgical considerations or on the economic limit of operation with heat resisting alloy muffles and retorts.

The second reason why equipment is critical is the increased emphasis on uniformity which arises when the process of carburising is speeded up by two or more times by increasing the temperature. This puts great emphasis on uniformity of heating and atmosphere distribution, and automatic control is necessary in order to secure satisfactory operation.

The sealed-quench type of unit is more suited than the pit furnace in most respects for this high temperature work, and both batch sealed-quench and pusher type units for greater outputs are now available for temperatures well above normal carburising levels. Operating experience with ceramic fans is proving most encouraging; increased production has now been arranged and more widespread employment is confidently anticipated. Indeed, development work employing these fans and ceramic heating tubes is now more concerned with the temperatures for such operations as high speed steel hardening, say, 1,300° C., rather than the lower level of temperature for high temperature carburising.

Field experience with those operating equipment on high temperature carburising has fully confirmed the ability of suitably designed units to perform at 1,000° C., and higher, without excess maintenance or operating problems. Some operators now have experience of up to two years on fully commercial operation of high temperature carburising, with excellent results from the maintenance viewpoint.

Atmosphere Control

Work continues at Ipsenlab at Rockford, U.S.A., with both the regular production use of high temperature carburising and experimental work on control of the atmosphere and other aspects. Ipsenlab is an independent corporation of Ipsen Industries, Inc., operating partly as a development centre, partly as a commercial heat treatment unit, and partly as a demonstration plant for Ipsen equipment. A considerable amount of development work is carried out on actual production furnaces in this way.

The majority of the furnaces at Ipsenlab are batch type controlled atmosphere units, with several furnaces supplied from a single atmosphere generator. This requires operation of the generator at a nominal dewpoint. An adjustment is made to the various furnaces with natural gas or air additions to obtain the required carbon potential. All of the furnaces are equipped for monitoring and recording of atmosphere dewpoints, with automatic control for selected units. In the same way the endothermic generators are provided with automatic dewpoint equipment which maintains a pre-set control dewpoint on the generated gas. Both Alnor and dewcup measurements are made occasionally for comparison and check-out of the existing control equipment.

Automatic control of furnace dewpoint is used for

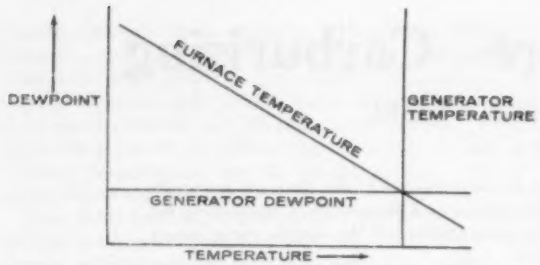


Fig. 1.—Furnace temperature effects on dewpoint

applications falling in four categories, all of which involve restrictive carbon ranges :—

(1) restoration of decarburised surface of precision castings (0·18–0·23% carbon range) ;
(2) homogeneous carburising or carbon restoration (0·40–0·50% and 0·60–0·70% carbon range) ;
(3) all carburising cycles ; and
(4) selected carbonitriding cycles.

The specification governing precision castings is very critical due to their highly stressed application, and the restrictive carbon limits are important because of subsequent welding operations. A control dewpoint of +65° F. is used. Since the standard supply of endothermic gas in the shop is at a lower dewpoint than this, actual control is accomplished by introducing air directly into the work chamber in excess of the required amount to bring the dewpoint up to 65° F. Natural gas additives introduced through a solenoid-operated by-pass system, controlled by an automatic dewpoint instrument, brings the dewpoint back to the +65° F. setting. This method has been found easier to control than direct modulation of the air additive.

At these low carbon contents, and in the medium carbon ranges, there is very little problem in maintaining reproducible results. The available charts on the carbon-dewpoint temperature correlation are reasonably accurate and can easily be adapted to the specific process, keeping in mind that compensation may be required for natural gas and ammonia additions, surface area of the work, tightness of the equipment, and other factors affecting atmosphere equilibrium reactions.

The basic relationship of generator-furnace dewpoints may be of some aid as a preliminary check-out of equipment. (Fig. 1). The dewpoint of the generator and of the furnace should be identical at a furnace operating temperature equal to that of the generator. If a generator operating at 1,000° C. is producing a +30° F. dewpoint atmosphere, a furnace operating at 1,000° C. with this same atmosphere should also register a +30° F. dewpoint. There must be equilibrium conditions in the furnace to maintain this relationship, and in practice it may require a considerable period to reach this condition. A persistent deviation, however, could indicate a furnace or generator problem, possibly incorrect temperature control of the furnace or generator, or excessive leakage at the furnace. This relationship may not hold true for all makes of equipment, as the critical factor remains the true reaction temperature of the generator. Relationship of the retort temperature to that registered by the control thermocouple of the generator must be determined before any compensation can be applied.

This relationship of dewpoint between furnace and generator becomes quite a factor in high temperature carburising. It is well known that dewpoint is inversely proportional to carbon potential, and for a given potential inversely proportional to temperature. The relationship on the basis of just temperature and dewpoint variables is not often considered, however. There is a natural tendency to add extra propane to lower the dewpoint, and since higher temperatures require lower dewpoints, it is logical to expect a considerable increase in hydrocarbon additions as the temperatures are moved up for higher carburising temperatures. Actually this reasoning is not entirely true, because as the temperature is increased the measured dewpoint will also change : a typical charted sequence for various furnace temperatures, while maintaining a constant atmosphere flow, might read as follows :—

Temperature	Dewpoint
815°C.	+57°F.
875°C.	+48°F.
925°C.	+39°F.
985°C.	+31°F.

(Generator operating at 985° C. with + 32° F. dewpoint)

Results of Test Runs

Fig. 2 pictures the carbon potential-dewpoint data applying to the temperature ranges that are considered practical for carburising. These curves are plotted from recent test data selected from sample processing and production runs. Previously reported information had indicated that a temperature of 1,035° C. might be practical for many applications, and several sample runs were made at this temperature. A normal 0·85–1·00% carbon specification would require dewpoint control within an estimated 2° F. range at this temperature. A comparative 4° F. deviation would be allowable at 925° C. This indicates the critical need for automatic control.

One such test series was run to evaluate the possibilities of elevated temperatures for carburising bearing retainers. Although this application would not be considered ideal because of dimensional tolerances, a review of the results may be enlightening. (Table I). The parts were carburised in an Ipsen TFC–150 furnace at 1,035° C. for three hours to produce an 0·040–0·050 in. effective case. Carbon step-down bars and shim stock

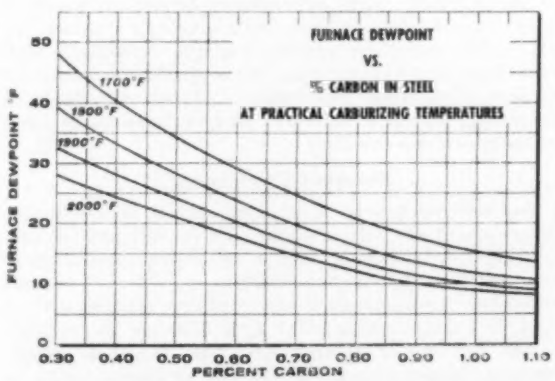


Fig. 2.—Relationship between furnace dewpoint and percentage carbon in steel at practical carburising temperatures.

TABLE I.—RESULTS OF TEST RUNS

Heat No.	Generator Dewpoint (°F.)	Natural Gas Addition (c.f.h.)	Dewpoint Temperature (°F.)			Carbon Content of Steel (%)
			Dewtronik	Alnor	Dewcup	
1	20 to 22°	3 (const.)	12 to 21°	13 to 20	8 to 20	0·72
2	16 to 20°	7 (const.)	−4 to +4	0 to 5	−8 to +4	1·48
3	24 to 27°	1–4, 1–5 (adjust)	11	9 to 11	8 to 13	1·13
4	20	2–5	12	10·5to14·5	3 to 5	0·96
5	18 to 20	2–5	12·5	11 to 14	6 to 8	0·90
6	20	2–5	13	11·5to13·5	8 to 16	0·87
7	20	2–5	11·5	9 to 14	10 to 13	1·00

*Not on automatic control

specimens were run with each load. The furnace was conditioned to the selected dewpoint before loading. Alnor and dewcup readings were made at frequent intervals in addition to record and control with an Ipsen Dewtronik dewpoint controller. The first three runs were preliminary attempts to check out previous cycles and establish natural gas additive ranges. Generator atmosphere was first supplied from a unit on manual control, and results indicate the value of positive dewpoint monitoring directly at the source, in addition to dewpoint control at the furnace for such critical applications. In heats No. 1 and No. 2, constant natural gas additions were used with no attempt to control dewpoints automatically at the furnace. Prior tests had indicated approximately 5 to 10 c.f.h. of natural gas would be required for proper control with a +30° F. dewpoint generator atmosphere.

In Table I, the values for carbon percentage were all obtained by step-down bar analysis, and indicate a natural sequence with respect to dewpoint control settings. These runs, although in progression, covered a period of two weeks, and were scheduled between production loads. The furnace was conditioned prior to each load. Fig. 3 shows an actual reproduction of a Dewtronik record chart for one of the runs given in Table I, the actual heat being No. 3.

In order to understand this, some brief comment on the operation of the Dewtronik instrument may be helpful. This instrument operates by measuring the dewpoint of the gas directly, and cools down a continuously flowing gas sample in contact with a sensing head until dew forms. Immediately this happens, the head is heated to remove the dew and as soon as the dew is removed, the head cools again until dew is formed. Hence a small oscillating temperature is produced in a thermocouple located in the sensing head. This temperature is recorded as a broadened line by a pen on the chart as shown in Fig. 3, on which the range of oscillation and the fluctuating control can readily be seen. The lower point of the band may be taken as the actual dewpoint. This instrument is set up so that the position of the measured dewpoint in relation to the control setting operates a solenoid valve which admits a minimum or maximum flow of additive gas to the furnace. This can be set to give any minimum and maximum required to suit the conditions, and as will be seen from the chart it was originally set in this cycle for the minimum of 1 c.f.h. and a maximum of 4 c.f.h. Towards the end of the run when the generator had drifted from +24° to +27° F. this quantity of additions was insufficient and the furnace dewpoint began to drift from control. The additive panel was then reset to give 1 to 5 c.f.h. thus bringing the unit back under control again. In this particular furnace an atmosphere flow in the order of 200 c.f.h. would be normal.

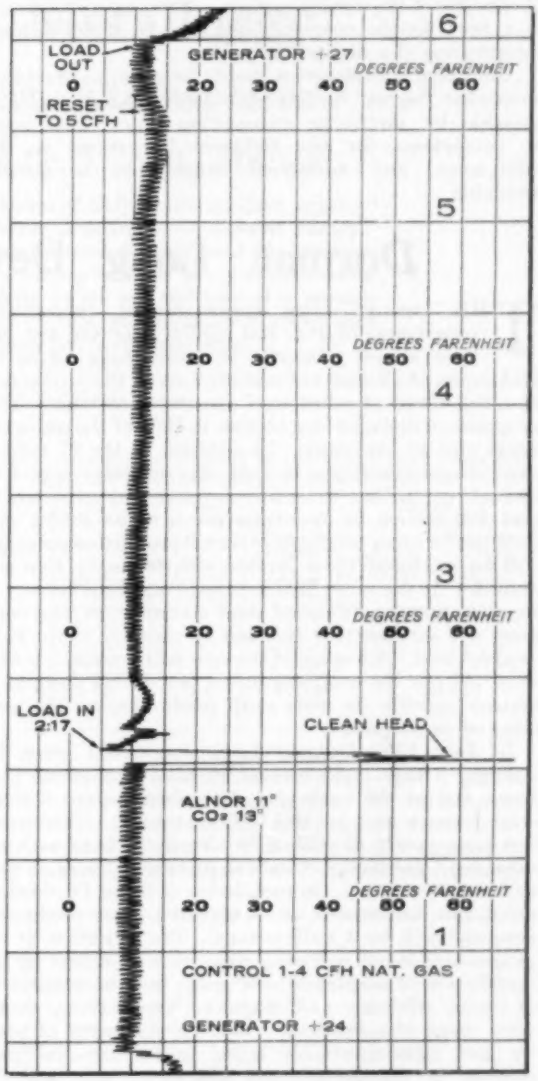


Fig. 3.—Reproduction of Dewtronik record chart for heat No. 3 [see Table I].

An examination of results would indicate some unreliability in manual dewpoint measurement as a basis of control, although frequent checks are encouraged. Gas analysis for each load would be impractical, but with a recording fractometer or other gas analysis equipment now available, positive correlation on a pilot basis could be very helpful. Investigations of this nature are being made on production heats, and it is hoped that related dewpoint to gas analysis and carbon analysis information on varied treatments will be available for release in due course. In this work a +20° F. generator carrier gas has been found most suitable for most cycles at high temperature.

One characteristic of dewpoint measurement at elevated temperatures is the necessity of very close attention to sample gas filtering and increased frequency

of cleaning of the sensing system. Fortunately, cleaning is a very simple operation and can be done without interrupting the carburising cycle.

Additional consideration should be given to providing somewhat higher furnace pressures than normal, if possible by increasing atmosphere flow. This helps to compensate for any tightness limitations on the equipment, and additional atmosphere is usually available.

It is hoped these notes on further practical work in this field will be of interest. It is already established that high temperature carburising is both practical and economical, at least for some types of carburising work. It would be regrettable to allow this possibly potent source of increased productivity and efficiency to be developed only abroad, and it is hoped the present interest here will steadily develop into practical application.

Dorman Long Development Programme

THE Dorman Long major capital expenditure programme of over £60 million since the war has been aimed primarily at establishing advanced techniques at all stages of manufacture in the production of a full range of rolled steel structural sections. This programme included completion in 1958 of the universal beam mill at Lackenby. In addition to the £7 million expenditure announced in July, the company is now to embark on further extensive development expected to cost £36 million to modernise much of its rolling mill facilities for other products. Overall production potential will be increased by a further 400,000 ingot tons per annum. At the same time as improving the efficiency of its plate and semi-finished steel manufacture, the company will substantially increase its capacity in the light product field. A feature of the new mill proposals is that they will put the company into a favourable position to embark quickly on wide strip production at the next stage of development.

In July 1960 Dorman Long announced plans for building a third 1,500-ton-a-day blast furnace at Clay Lane, and at the Lackenby steel plant a sixth 360-ton steel furnace and a third 600-ton mixer. This work, involving nearly £7 million, is already in hand and will raise ingot production from the present 2·3 million tons to 2·6 million tons. On completion of Stage IV development, now announced, the company's ingot production potential will be 3 million tons. The objective of the programme is not primarily expansion of output by the installation of additional new units, but the attainment of higher efficiency and, therefore, productivity over a wide range of operations. While replacement of plant by new units inevitably raises production, the main increase in finished outputs will be in light and flat products where a rising national demand is expected.

While Stage IV developments are a balanced scheme as a whole, many individual projects are involved, and it is essential that the bringing into production of the various units should be carefully phased. The initial part of the programme includes a new primary mill at Lackenby for the production of 22,500 tons of slabs and blooms per week from ingots of up to 20 tons in weight. At Lackenby will be built also a new high capacity universal plate mill to replace the existing universal plate mill at Redcar—the only one in the United Kingdom. Universal plate up to 42 in. wide has been a speciality of Dorman Long for more than forty years, and the new installation will give a major advance in the efficiency of production and allow for an increase in width and improved quality.

A new wire rod mill of high capacity to roll coiled bar from $\frac{1}{4}$ in. diameter down to 5 gauge will be constructed at Lackenby, and the existing rod, bar and narrow strip mill adjoining the Lackenby beam mill will be modified by the provision of additional cooling facilities and extra stands for the production of larger diameter bars (over $\frac{3}{4}$ in. dia.) in coil and straight lengths. Present facilities for rolling narrow strip up to 12 in. wide at this mill will be retained, and provision will be made for pickling, oiling and cold rolling of this strip, thus taking the company's products in this developing field to a more highly finished stage. To provide the increased billet requirements arising from the installation of the new rod mill, it is proposed to add to the semi-continuous billet facilities recently completed at Lackenby.

A start will be made immediately on these earlier projects in the programme coupled with the necessary ancillary services. After completion there should be a steady build-up of rolling rate on these new units but full outputs will take some time to achieve. Projects in the Stage IV programme concerned with increasing iron and steel making capacity will be phased in later to coincide with this expansion of new mill requirements, as will other mill schemes necessary for efficient overall production in line with the objects of the Stage IV development programme.

The Iron and Steel Board have approved in principle the general objectives of the Stage IV development programme and formally agreed the details of the projects to be implemented in the early phases costing approximately £24 million.

The layout of the new slabbing mill and universal plate mill at Lackenby will be arranged for extension later into a medium width strip mill, possibly 48 in. wide. A combination mill for rolling universal plate and strip is technically attractive. In due course the company's present mechanised production of sheets will need to be replaced and, therefore, the company intends as part of a Stage V major plant development to put forward proposals at the earliest appropriate date to proceed with the addition of hot strip finishing stands to the universal plate mill and appropriate cold rolling facilities.

Ferroclad Bricks

GENERAL Refractories, Ltd., the sole licensees for the manufacture and sale in Great Britain of metal-cased basic bricks produced under the Heuer patents and sold by them under the trade name Ferroclad, have decided that in view of the substantial increase in the demand for these bricks, especially in connection with the development of the all-basic furnace, it is in the national interest that these patents should be made available to other manufacturers. Accordingly, arrangements have now been made whereby Pickford Holland & Co., Ltd., will be entitled to manufacture metal-cased bricks under these processes and to market them under their own name of Metalclad.

Low Pressure Metallurgy

Vacuum as an Inert Atmosphere

By A. S. Darling, B.Sc.(Eng.), Ph.D., A.M.I.Mech.E.

In the October, November and December, 1959, issues of METALLURGIA, *there appeared an article by Dr. Darling entitled: "Low Pressure Metallurgy—A General Survey." The present article is the second under the same general heading, and the reference numbers follow on those in the earlier article. In dealing with the use of vacuum as an inert atmosphere, the author discusses such aspects as the gas tightness of a vacuum system; contamination during vacuum melting of such metals as iron, nickel and uranium; and vacuum oxidation processes in the solid state.*

(*Continued from page* 142 *of the October issue*)

Contamination during Vacuum Melting

METALLIC oxides are in general so stable that dissociation pressures are well below the oxidising potential of the best attainable vacuum systems, as can be seen from Fig. 4. Many molten metals are capable of dissolving oxygen in atomic form at concentrations lower than those at which a discrete oxide phase can exist. Oxygen can only be removed from such solutions, which are thermodynamically more stable than the oxide, with the greatest difficulty.

Normal vacuum melting practice does not usually permit the melt to come into equilibrium with the furnace atmosphere. Since oxygen in solution is uniformly distributed through the melt, oxygen concentrations are frequently very much lower than would be suggested by the degree of vacuum.

Oxidation of Nickel and Iron during Vacuum Melting

Molten iron and nickel are both capable of dissolving considerable quantities of oxygen and this solubility increases with rise of temperature. Table II summarises the oxygen solubility and dissociation pressures of the saturated solutions over the relevant temperature ranges. The essential conclusions are that nickel, at a given temperature, is capable of dissolving approximately 4 times as much oxygen as iron and that the dissociation pressure of the saturated solutions of oxygen in nickel are greater by several orders of magnitude than those of iron. At 1,700° C. for example, the calculated dissociation pressure of a saturated solution of oxygen in equilibrium with nickel oxide is 2·3 mm. Hg. On thermodynamic grounds such a solution should decompose during normal vacuum melting processes, whereas the oxygen content of iron under similar conditions should increase considerably.

The experimental verification of such predictions is complicated by factors such as differences in the initial carbon and oxygen contents of the metals investigated, and by varying degrees of metal/crucible reaction. Table III summarises some experimental data relating to nickel which

TABLE II.—MAXIMUM SOLUBILITY OF OXYGEN IN IRON ([135, 137]) AND NICKEL ([135]) AT TEMPERATURES FROM 1,450–1,750° C.

Temp. °C.	Iron			Nickel	
	Max. Solubility (Wt. %)		Dissociation Pressure of Saturated Solution (mm. Hg.)	Max. Solubility (Wt. %)	Dissociation Pressure of Saturated Solution (mm. Hg.)
	Chipman (136)	Gokcen (137)			
1,450 ..				0·28	$2·1 \times 10^{-3}$
1,500 ..				0·4	$5·4 \times 10^{-3}$
1,550 ..	0·184	0·190	$1·9 \times 10^{-3}$	0·58	$1·6 \times 10^{-2}$
1,600 ..	0·227	0·231	$4·5 \times 10^{-3}$	0·82	$4·0 \times 10^{-2}$
1,650 ..	0·280	0·278	$1·0 \times 10^{-2}$	1·15	0·96
1,700 ..	0·338	0·331	$2·0 \times 10^{-2}$	1·59	2·3
1,750 ..	0·408	0·391	$4·1 \times 10^{-2}$		

was published by Stauffer several years ago[139]. These results confirm in a striking manner the square root dependence between solubility and pressure which exists in unsaturated solutions. This relationship appears to apply up to oxygen contents of 0·10%, nitrogen contents

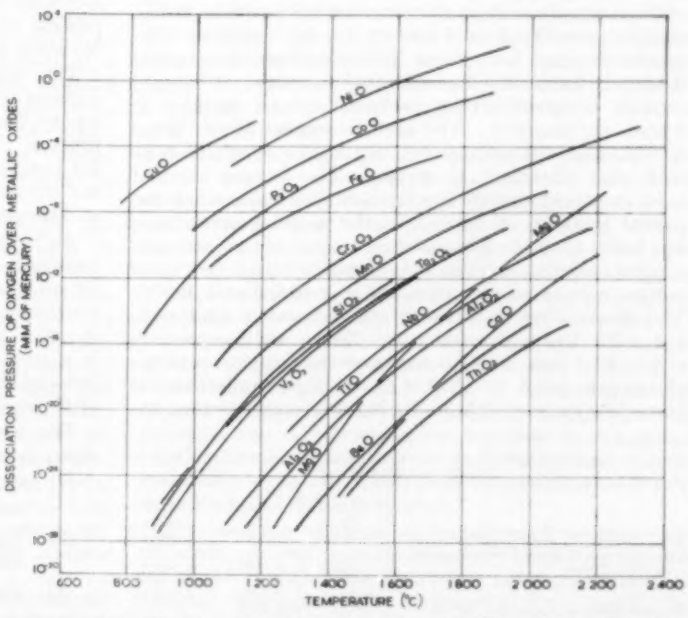


Fig. 4.—Dissociation pressures of some metallic oxides. Oxygen pressure in mm. Hg.

TABLE III.—GASEOUS CONTENT OF MOLTEN NICKEL EXPOSED TO GAS AT VARIOUS PRESSURES FOR 10 MINUTES
(Percentage composition of starting material: oxygen, 1×10^{-7}; nitrogen, 1×10^{-5}; hydrogen, 3×10^{-5}.)

Oxygen Pressure (mm. Hg.)	Oxygen in Nickel (%)	Nitrogen Pressure (mm. Hg.)	Nitrogen in Nickel (%)	Hydrogen Pressure (mm. Hg.)	Hydrogen in Nickel (%)
0·001 ..	$1{\cdot}5 \times 10^{-3}$	27	$1{\cdot}3 \times 10^{-4}$	2·2	7×10^{-5}
0·02 ..	$4{\cdot}0 \times 10^{-3}$	50	$3{\cdot}0 \times 10^{-4}$	6·7	22×10^{-5}
0·10 ..	$15{\cdot}0 \times 10^{-3}$	760	$8{\cdot}0 \times 10^{-4}$	33·2	34×10^{-5}

TABLE IV.—GAS CONTENT OF ARMCO IRON AFTER MELTING IN VARYING DEGREES OF VACUUM

	Oxygen (%)	Nitrogen (%)	Hydrogen (%)	Carbon (%)
As received	0·0517	0·008	0·0005	0·05-0·08
Melted at 4 mm. Hg. ..	0·0309	0·0079	0·00015	0·0082
Melted at 5×10^{-2} mm. Hg.	0·0443	0·0067		0·0056
Melted at 5×10^{-3} mm. Hg.	0·0455	0·0083	0·00012	0·0064
Melted at 5×10^{-4} mm. Hg.	0·0444	0·0071		0·005

of $3{\cdot}0 \times 10^{-4}$% and hydrogen contents of 34×10^{-5}%. The fact that such solubilities were attained within 10 minutes indicates a rapid rate of diffusion in the liquid state.

The results in Table III are inadequate, insofar as no reference is made to carbon content of the starting material. A recent paper of Pfeiffer[140] is more explicit in this connection. This investigator studied the effect of differing vacuum melting conditions on the final gaseous and carbon contents of nickel and iron. The results demonstrated the ability of a small residual carbon content to maintain the final oxygen content at a value much lower than calculations based entirely upon vacuum conditions would predict. Table IV summarises data obtained on Armco iron having an initial carbon content of 0·06%.

The data indicate that in this instance the effect of degree of vacuum is negligible compared with the initial reducing effect of residual carbon. At 1,600° C., for example, with no carbon present, the melt might have been expected to come into equilibrium with an air pressure of $2{\cdot}25 \times 10^{-5}$ mm. Hg by absorbing 0·227% of oxygen. Due to the carbon content, however, vacuum melting at 4 mm. Hg produced an oxygen content of only 0·03%. Pfeiffer's results do not refer to the complicating effects of metal/crucible reactions which must undoubtedly have accounted for the virtually constant oxygen content in the iron under all vacuum melting conditions.

Experiments with nickel gave similar results. 20 kg. charges were held at 1,500° C. for 15 minutes. The results obtained with Mond Nickel shot are summarised below in Table V. Considerable increases of oxygen content occurred during air melting and melting at 4 mm. Hg pressure. The carbon content of the Mond Nickel shot was presumably not high enough to cope with this quantity of oxygen. The oxygen content even increased slightly above the initial value when the partial pressure of oxygen in the melting atmosphere was lower than the dissociation pressure of the saturated oxygen solution. Table II indicates that 0·4% of oxygen can be taken into solution in nickel at 1,500° C. The dissociation pressure of this saturated solution is $5{\cdot}4 \times 10^{-2}$ mm. Hg of oxygen. Now a melting pressure of 5×10^{-2} mm. of Hg corresponds to a partial pressure of oxygen of 1×10^{-2} mm. of Hg. Application of Sieverts square root law of solubility suggests that the equilibrium solubility of oxygen in nickel at 1,500° C. under an air pressure of 5×10^{-2} mm. Hg should be 0·17%. This value is approximately twice as high as that actually determined. The small increase in oxygen content over the initial value probably indicates a gradual tendency to increase in the direction of the equilibrium value. The same basis of calculation suggests that the equilibrium oxygen content in a vacuum of 5×10^{-4} mm. Hg would be 0·017%. The actual oxygen content determined was 0·0058%—approximately one third of that predicted. The slight increase over the initial oxygen value probably indicates in this instance also a gradual tendency towards movement in the direction of equilibrium conditions. The nitrogen results are particularly interesting. Whereas the nitrogen content of the nickel decreases with melting pressure, pressure has little effect upon the nitrogen content of iron. Dilute solutions of nitrogen in iron are of course comparatively stable.

TABLE V.—GAS CONTENT OF MOND NICKEL SHOT AFTER VARIOUS TYPES OF TREATMENT

	Oxygen (%)	Nitrogen (%)	Hydrogen (%)
Mond Nickel shot, as received—unabraded	0·00448	0·0005	0·00015
Mond Nickel shot abraded to remove surface oxide films	0·00382	0·00053	0·00029
Open melt	0·265	0·017	0·000012
Open melt	0·225	0·0147	0·000013
Vacuum melted at 4 mm. Hg.	0·131	0·0097	
Vacuum melted at 5×10^{-2} mm. Hg. ..	0·0077	0·0014	0·00001
Vacuum melted at 5×10^{-4} mm. Hg. ..	0·0058	0·00053	0·00006

Contamination of Pure Uranium during Vacuum Melting

Uranium forms an oxide many times more stable than that of iron and the difficulties encountered during vacuum melting are considerably greater. There appears, even in the liquid state, to be no range of oxygen solubility, with the result that tenacious oxide skins invariably cover the surface of melted metal particles and retard their agglomeration. Reliable density values for high purity uranium did not, in fact, become available until 1955, when details of a suitable melting process were published by Blumenthal[141] of the Argonne National Laboratory.

The essential problem was to melt into massive form, without contamination, needle-like crystals of high purity uranium produced by a fused salt electrolytic process. Vacuum melting was found to be necessary for removal of the large quantities of gas dissolved in the crystals and for distillation of contaminants such as lithium and potassium.

An interesting conclusion of this research was that a resistance furnace was better than induction heating for maintaining a high state of purity in the melt. This conclusion would undoubtedly apply to the melting of other metals where the maintenance of high purity is a primary requirement. The stirring action of the induced currents increased the rate of contamination of the melt from crucible, slag, and furnace atmosphere.

The essential stages involved in the production of clean uranium ingots were as follows :—

(1) Satisfactory preparation of crystals before melting.

(2) Rapid vacuum melting under a pressure at no stage exceeding 10^{-4} mm. Hg.

(3) Directional solidification.

Surface preparation of the crystals involved preliminary treatment with dilute nitric acid to remove oxide

films, followed by several stages of washing, pickling, rewashing, and drying in absolute alcohol. The crystals were frequently dentritic in form and quantities of fused electrolyte were often trapped in small cavities. This electrolyte was richer in carbon than the crystals, which had a very low carbon content. For this reason the final analytical results showed a strong correlation between the carbon content of the ingots and the thoroughness of the washing treatment carried out on the crystals. The best results were achieved when the first washing, carried out in running tap water, was prolonged for 48 hours.

The extent to which surface oxide had been removed by the acid treatment was revealed in a sensitive manner by the interference colours on the surface of the vacuum dried needles. A slight yellowish tinge was satisfactory, whereas a greenish yellow colour indicated an oxide skin heavy enough to interfere with the subsequent melting operation.

Melting was carried out in urania crucibles. Molten uranium does not wet urania and appreciable reaction between the two materials does not occur at temperatures below 1,700° C. The urania crucibles lasted for four or five melts: a slight undercut which developed near the top of the melt eventually made it difficult to remove the solidified ingot.

The crucible was suspended in a special cage at the centre of a vertical closed-end refractory (Mc.Danel) tube. This cage was manufactured from a specially prepared tantalum-tungsten alloy and was in turn suspended from a vertical alumina tube. Attachment between the alumina tube and metal cage was effected with a sapphire cotter pin. At no time did the metal cage come into contact with the refractory tube, which was evacuated at its upper end by a high speed pumping system, comprising a conventional diffusion, booster and forepump combination.

During the earlier stages of the research, back streaming from the diffusion pump resulted in excessive carbon pick-up by the molten metal, and a tantulum cup completely surrounding the crucible was found to be necessary. In order to eliminate such contamination a high speed "Chevron type" cold trap was evolved and fitted between furnace and diffusion pump. The effectiveness of this trap was demonstrated by subsequent analysis of the experimental results, which showed no correlation between carbon content and the use of a tantalum protection shield.

After assembly of charge, crucible and cage, the furnace was pumped down cold to a pressure of 5×10^{-7} mm. Hg. A pressure maximum of 1×10^{-4} mm. Hg which occurred during the initial heating period was attributable to gas release from the refractories. In order to avoid undue oxidation it was necessary to heat rapidly and reduce the time during which large areas of metal were exposed to oxidation. Slow heating did not produce satisfactory ingots. The individual particles did not fuse effectively together due to stable oxide skins upon their surfaces. Satisfactory melting procedure involved the heating of 300–800 g. of metal to 1,200° C. in 45–50 minutes. The metal was held at this temperature for 3 minutes, after which the crucible was lowered to the cold part of the furnace at a rate of 13 in. per hour.

High purity uranium ingots produced in this manner contained 15 p.p.m. of carbon, 8 p.p.m. of oxygen and less than 10 p.p.m. of nitrogen. Spectrographic and chemical analysis indicated that the metal was better than 99·993% pure. The density, determined by displacement in carbon tetrochloride, was 19·050 ± 0·002 g./cu. cm.

In spite of the precautions observed in this work the resultant ingots were not completely clean from the metallographic point of view. Numerous minute inclusions, believed to be particles of residual fused salt electrolyte, could be observed in polished, unetched sections, and closer examination revealed the presence of a second phase. This phase appeared as an incomplete eutectoid network which did not follow the boundaries of the *alpha* grains.

The fused salt inclusions could be practically eliminated by liquation at 1,200° C. for 24 hours, but the eutectoid network was very much more stable. Its incidence was found to be minimised by the speed of melting and the precipitate was concluded to be caused by discrete particles of carbide, nitride and oxide. 10 p.p.m. by weight of carbon are equivalent to 291 p.p.m. by volume of uranium carbide, and similar ratios are applicable to the oxide and nitride. Metallographic examination revealed, therefore, very small quantities of such contamination. The precipitate could be taken into solution by quenching from 1,000° C. Subsequent ageing for long periods at 600° C. in the high *alpha* range caused reprecipitation.

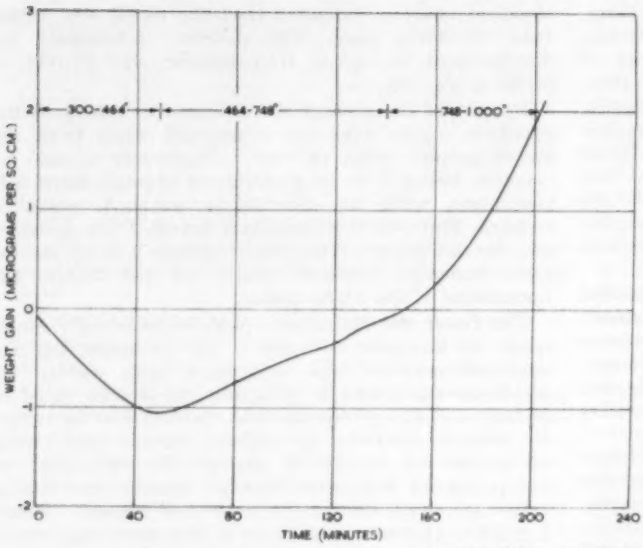
Such results emphasise the extremely low solubility of carbon, oxygen and nitrogen in pure uranium.

Vacuum Oxidation Processes in the Solid State

The best vacuum systems available are capable of producing pressures of the order of 10^{-13} — 10^{-14} atmospheres[143]. The oxygen potential of such systems is higher than the dissociation pressure of chromic oxide, for example, which at 850° C. is only 10^{-23} mm. Hg. It is obvious therefore, that even under the best possible conditions, oxidation *in vacuo* cannot be prevented. The quality of the vacuum can only be assessed according to its ability to slow down the rate of oxidation. Oxidation in the solid state is perhaps of more importance than the solution of oxygen in liquid metal, since the conditions ensure the formation of surface layers of very high oxygen content.

Numerous sintering and annealing processes are carried out *in vacuo*, and it is of interest to attempt some account of the relationship between vacuum quality and degree of gaseous contamination. One of the very early papers on the fabrication of titanium[143] afforded a good illustration of the gaseous contamination which occurs, even under fairly good vacuum conditions. Hot and cold rolled titanium sheet was annealed in a vacuum of the order of 5×10^{-4} mm. Hg. Minimum yield strength and hardness values were achieved by annealing between 700 and 900° C. Annealing temperatures of 1,000–1,200° C. produced an increase in tensile strength and a corresponding decrease in elongation. Similar results were experienced during the powder metallurgy operations: compacts sintered above 1,000° C. were hard and difficult to work.

The vacuum reactions of metals such as zirconium, titanium, niobium and tantalum are of general interest. All these metals form extensive solid solutions with oxygen, nitrogen and hydrogen. The oxides and nitrides of titanium and zirconium are particularly stable, while those of niobium and tantalum are less so,



(*Courtesy of Journal of Metals*)

Fig. 5.—Weight changes observed during the heating of niobium between 300° and 1,000° C. in a vacuum better than 10^{-6} mm. Hg. (Gulbransen and Andrews.)

thus permitting some degree of purification under high vacuum conditions. In view of the industrial importance of such metals, numerous studies of the kinetics of their reactions with the gas phase have been undertaken, leading to a more complete understanding of their behaviour under high temperature conditions.

Gulbransen and Andrew found the vacuum microbalance a convenient tool for such kinetic studies[144-149]. This type of equipment permitted the measurement of weight changes as small as 0·3 μg. The vacuum system was of Pyrex glass which was sealed directly to a double walled zircon furnace tube. The leak rate after overnight pumping at 900° C. was 5×10^{-3} micron litres per hour. Residual pressures at 1,175° C. were better than 10^{-6} mm. Hg. Even under such clean conditions contamination occurred when the metals were rapidly heated in vacuum. The formation of surface films on specimens at room temperature was minimised by carrying out the last stages of abrasion under purified kerosene. Fig. 5 illustrates the behaviour of niobium on heating between 300° and 1,000° C. in a vacuum better than 10^{-6} mm. Hg. The weight change in micrograms is plotted against the time. During the initial period of the reaction, between 300 and 464° C., gas was evolved, and the specimen lost weight. Above 464° C. a gain in weight was observed because of the gettering action. Gulbransen concluded that over this portion of the temperature range the surface of the specimen was probably film free and the reaction rate was not limited by surface barriers. Zirconium exhibited the same type of behaviour. The extent of the reaction with tantalum was considerably lower. It was concluded that heating to reaction temperature under the worst conditions of time and temperature produced on all four metals a surface film less than 50 Angström units thick.

McMillan and Gulbransen described equipment and techniques for evaluating the contamination potential of high vacuum furnaces in 1955[149]. It had hitherto been usual in the Westinghouse Research Laboratories to assess the oxidising potential of a high vacuum furnace by heating a small strip of metal and assessing the thickness and colour of the surface oxide films. Such methods were not completely adequate, because a minimum of several hundred Angström units of oxide was required before surface films became visible, and absolute pressure determinations were not a true indication of the reactive potential. In order to evolve a more quantitative approach to the problem, these investigators applied electron optical methods to study the oxidation reactions occurring in high vacuum. Previous to this, Bardolle and Bénard used optical microscopic methods for studying the oxidation of Armco iron in vacuums of the order of 10^{-3} to 10^{-2} mm. Hg at 700–850° C.[150] Under such conditions well oriented oxide nuclei could be detected.

Iron

Gulbransen's studies demonstrated that electron micrographic techniques enabled oxide films 10–25 Angströms thick to be readily observed. With Puron and Armco iron in high vacuum, the following reactions were found to occur: (1) the direct oxidation of iron to FeO; (2) the solution of oxygen as FeO in the metal; and (3) the reaction of carbon in the metal with either dissolved or combined oxygen to form carbon monoxide. Since the equilibrium ratio of H_2O/H_2 over iron and iron oxide at 850° C. is $10^{-0.17}$ the reaction of water vapour with the metal to form FeO and hydrogen was not observed.

The experimental equipment employed had a limiting pressure of about 10^{-7} mm. Hg at 800° C. A vacuum tight mullite ceramic tube was sealed to Pyrex glass tubing which permitted direct connection to the high speed mercury diffusion pumps. Dry ice, and liquid nitrogen traps prevented diffusion of mercury into the system. Three gauges were employed: a sensitive McLeod gauge permitted direct measurement of pressures down to 10^{-5} mm. Hg; a cold cathode ionisation gauge of the Philips type operated from 0·7 mm. to 10^{-7} mm. Hg; and a Bayard-Alpert gauge recorded pressures from 10^{-5} to 10^{-8} mm. Hg. At 800° C. a vacuum of 3×10^{-7} mm. Hg was observed on the Philips gauge.

The equipment was designed so as to permit the admission of controlled quantities of highly purified oxygen or hydrogen. The specimens, supported on short sections of platinum wire, were moved in and out of the furnace magnetically. This permitted preliminary treatment of the specimens as a group or individual reaction without opening the system to the air.

The Puron specimens contained 0·005% of carbon and 0·04% of oxygen, no other impurities exceeding 0·0015%. The Armco iron was less pure: its carbon content was 0·015% and considerable quantities of sulphur, manganese, silicon and phosphorus were present. The oxide crystallites found under various experimental conditions were examined by optical and electron microscopy, and electron diffraction was used to study the crystal structure of the oxide film.

After up to 24 hours at 850° C. in a vacuum of 10^{-7} mm. Hg, both the Armco and Puron specimens appeared perfectly bright. Electron micrographic examination disclosed that oxidation had occurred in both instances. A thin surface film was present together with small randomly distributed crystallites. The crystallites on Puron iron were approximately 0·4 microns across after 3 hours of heat treatment, while those on the Armco iron were approximately one-tenth this size. Differences in the total quantities of oxide present in each instance were accounted for by the greater carbon content of the Armco iron.

The essential feature of the tests however was that at 10^{-7} mm. Hg the oxide film was randomly oriented with respect to the surface and the oxide particles had no definite crystal habit. After oxidation at 10^{-5} mm. Hg, however, the crystal habit was more definite and the oxide film was definitely oriented with respect to the metal crystals.

Further tests on Puron iron were made with a vacuum micro-balance. After prior heat treatment at pressures lower than 10^{-7} mm. Hg, specimens were reacted with small quantities of oxygen at low pressures. Oxidation was complete in one minute: after complete reaction with oxygen at 5 microns, the average film thickness was 453 Angström units. The electron diffraction pattern showed an oriented Fe_3O_4 structure.

Prior reduction treatment in hydrogen affected the subsequent formation of oxides in a pronounced manner. After reduction in normal unpurified hydrogen, specimens were reacted with oxygen at 1 micron. Both the crystallite habit and film orientation were then dependent upon underlying crystal orientation. This dependence was much less pronounced when the prior reaction was carried out with highly purified hydrogen.

Experiments made with Armco iron confirmed the pronounced effect of small quantities of carbon upon the surface reactions. Micro-balance work showed weight losses when specimens were held in high vacuum. This was due to the reaction between carbon and surface oxygen leading to the evolution of carbon monoxide. The magnitude of this reaction appeared to depend upon crystal orientation. Whereas reaction with oxygen at pressures of the order of 1–5 microns left Puron with a coloured surface film, the small quantity of carbon in Armco iron was sufficient to ensure a metal surface which was almost bright.

It was concluded that heat treatment in unpurified hydrogen aligned completely the defect structure of the metal which was responsible for oxide nucleation. This tendency was very much less pronounced when highly purified hydrogen was employed. Prior treatment in vacuum at low pressure induced no alignment of defects. The rate of oxidation appeared to vary proportionately with the oxygen pressure. Armco iron, oxidised at 2 microns increased in weight at a rate of approximately $1 \cdot 1 \times 10^{-6}$ g./sq. cm./min. The corresponding rate at 4 microns was $2 \cdot 07 \times 10^{-6}$ g./sq. cm./min.

Tungsten

Hashimoto, Tanaka, Yoda and Araki[151] have recently commented on the formation of needle crystals of tungsten oxide at pressure of 7×10^{-2} to 4×10^{-4} mm. Hg. Over the temperature range 700–1,000° C. needle- or rod-like crystals of several ten or hundred Angströms in thickness grew successively on the surface of tungsten wire. Electron diffraction studies indicated that the crystals were of γ tungsten oxide having the formula of $WO_{2 \cdot 72}$. The needle axes were parallel to the b axis of the monoclinic system. The electron diffraction pattern corresponded to photographs of single crystals oscillated up to 30° around the crystal axis. The evidence was interpreted as indicating that the needles consisted of a bundle structure of micro-crystalline needles, the orientations of which were distributed throughout a given angular range. This conclusion was confirmed by observation of the shape assumed by the needles after partial evaporation at about 1,100° C. at pressures varying from $3 \times 10^{-3} - 4 \times 10^{-4}$ mm. Hg.

The crystal growth process observed during intermittent heating was particularly interesting. During the first heating cycle in the range 700–1,000° C. very thin crystals grew. The thickness of these crystals increased during the second heating; this thickening process being accompanied by the simultaneous growth of a new needle at the crystal tips.

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New Liquid Helium Plant

BRITAIN'S first liquid helium plant to be run by an industrial organization started service recently at the Morden, Surrey, works of British Oxygen Research and Development, Ltd. The plant will produce sufficient liquid helium to meet all present demands from industry and research bodies in this country. The National Physical Laboratory, Teddington, which has run a plant for a number of years, is now gradually discontinuing production and handing over its customers to B.O.R.A.D. The only other sources of liquid helium in the U.K. are certain University laboratories, where low temperature research is carried out.

Soaking Pits Contract

THE first multi-zone, fully air-circulated, radiant tube soaking pits in this country have been ordered from Stordy Engineering, Ltd., by James Booth Aluminium, Ltd., for installation in their Kitts Green Works, Birmingham, where they will be used in conjunction with the 148-inch, four-high, hot reversing mill. Each of the two pits, will have a capacity of 60 tons of aluminium and the working temperature will be up to 620°C. The fully air circulated design has been specially developed by the manufacturers, and the radiant heating tubes will be fired by Stordy-Hauck oil burners.

The Spanish Steel Industry

A Recent National Financial Survey Summarised

Part of the national plant at Avilés, seen from the air during construction.

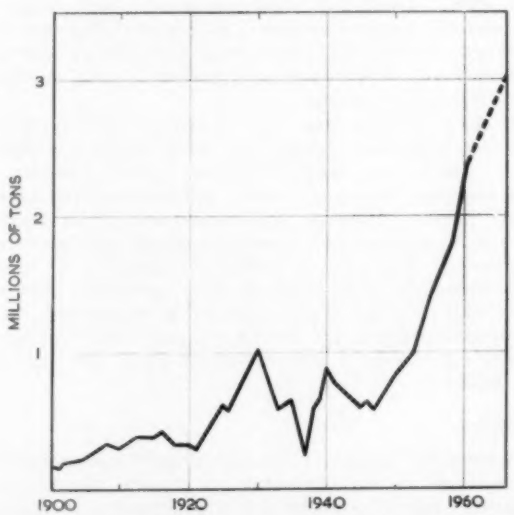
AN interesting feature of the Spanish steel industry is that as far back as 1929 it had already achieved the remarkable output of one million tons per annum, but this notable achievement was followed by a catastrophic falling-off in demand, so that this volume of production was not again achieved until 1954. It would be easy to criticise the economic planning (or lack of it) which resulted in building up a yearly capacity of one million tons without a suspicion that a few years afterwards the demand would drop to 500,000 tons, thus rendering nugatory all the costly extensions undertaken. In this connection, however, it is only fair to consider the difficulties encountered in the supply of raw materials. In the case of scrap alone, for the peak year of 1929 some 560,000/580,000 tons were used (of which 210,000 tons were imported) giving a scrap-steel relationship of 55/57%. In the period 1941–1945, the same plant, now a decade and a half older and consequently of diminished capacity, used only an average of 300,000 tons of scrap per annum, the average yearly imports being 15,000 tons.

A difference amounting to nearly 200,000 tons in the amount of imported scrap would in itself account for a drop in steel to 650,000 tons, which was the average for five years; in fact, due to the recurrence of scrap in the production cycle (the first steel manufactured results in turn in fresh scrap from rolling and machining, and so on), 1 ton of scrap makes possible the production of 5 tons of steel.

With the stagnation of the thirties overcome, the industry produced in 1958 1·53 million tons, 50% in excess of the once-famous peak of 1929, this output being contributed by eight integrated plants, a further four working on coal, forty-one undertakings producing and rolling steel, and sixty-nine devoted entirely to the production of steel, usually by electric furnaces, for casting and forging. Of the first-mentioned eight

TABLE I—RELATIONSHIP BETWEEN PRODUCTION OF STEEL AND TOTAL ELECTRICAL ENERGY, CEMENT, AND SULPHURIC ACID IN 1955

	Relationship between Steel and		
	Total Electrical Energy	Cement	Sulphuric Acid
World	100	100	100
Spain	67	25	17
U.S.A.	100	182	86
United Kingdom	104	126	117
France	149	93	106
Belgium and Luxemburg	312	151	115
West Germany	157	91	104
Italy	138	40	35
Austria	154	80	—
Hungary	128	110	—
Poland	79	104	121
Norway	12	16	25
Denmark	28	15	280
Finland	31	15	18
Turkey	32	18	130



Graph showing Spanish steel production since 1900 with projected production for 1960–65

plants, those of Altos Hornos in Bilbao and Sagunta, and the Empresa Nacional Siderúrgca, in Avilés, are the most important.

Many attempts have been made to estimate the future Spanish demand for steel. By the most usual method of evaluation—weight of steel consumed per head of the population—demand is low compared with other countries, the 1956 figures being Spain 50 kg., Italy 120 kg., France 276 kg., Germany 417 kg., and United States 600 kg. It is considered that a better method of expressing consumption is in relation to the demand for other essential supplies, such as total electrical energy, cement and sulphuric acid, as shown in Table I. It will be seen, however, that by this method also the Spanish index is very low, even allowing for the fact that the figures in Table I require adjustment for external trade: Belgium, for instance, exports 70% of her steel production. Probably the soundest comparison is with electricity, on which basis it can be argued that in 1955 a 50% greater production was necessary in order to establish an equilibrium. The shortage compared with cement is also noteworthy, as also the close resemblance between the figures for Spain and Italy. Compared with electricity, which makes the least unfavourable showing, the estimated annual deficit since the Spanish civil war has been of the order of 150,000 tons, or 100,000 million pesetas which has failed to enter the Spanish economy during this period. The scrap necessary to prevent this deficit (assuming that this shortage was the only cause) would have had a value in the order of 1,800 million pesetas.

Aerial view of the Baracaldo plant of Altos Hornos de Vizcaya, S.A.

Amongst the numerous forecasts of future demand which are available, the soundest appears to be that recently made by the commission set up by the Ministry of Industry to study the Spanish steel situation. This is based upon the relationship between the demand for electrical energy and for steel, and is one of the most moderate put forward. The conclusions of the commission in this respect are given in Table II. At the time the report was submitted, it appeared unlikely that the 2,374 million tons would be obtainable in 1960, but in 1962—a year which was the object of special study by the commission—it was estimated that there would be a probable production capacity of 3,990 million tons for a probable demand of 2,620 millions if the new extensions under way are completed to time.

TABLE II—ESTIMATED DEMAND FOR STEEL FOR THE YEARS 1960–1965

Year	Demand (thousands of tons)
1960	2,374
1961	2,494
1962	2,620
1963	2,751
1964	2,889
1965	3,033

In banking circles in Bilbao, however, it is considered that it will be one or two years later before equilibrium is established between demand and supply.

To achieve the estimated productions, it is of course necessary that the essential raw materials should be available, and 1962 in this respect would present the following picture. There should be no difficulty in connection with ore, of which considerable quantities are now being exported. It will be necessary to import coking coal to the extent of 850,000 tons per annum, but there should be no difficulties with other types of coal for metallurgical purposes, neither is it considered there should be any serious trouble with ferro-alloys which have been a problem in the past, although it will be necessary to import up to 80,000 tons of manganese. Scrap will also have to be imported to the extent of 200,000 tons during the year 1962. To show the vital necessity of these imports, failing such supplies, steel production for the year in question can be only 1,330 million tons instead of 2,620 millions.

Many tendencies in the world steel situation will have an influence upon the type and volume of Spanish production, especially in connection with the more direct use of low-grade minerals, some of which are undoubtedly destined to produce steels with qualities equal to those based upon scrap. The use of the oxygen-blown converter is now normal, not merely due to its lower installation cost per ton of capacity, and plants of this type are being installed both by the Sociedad Altos Hornos and by the national undertaking in Avilés.

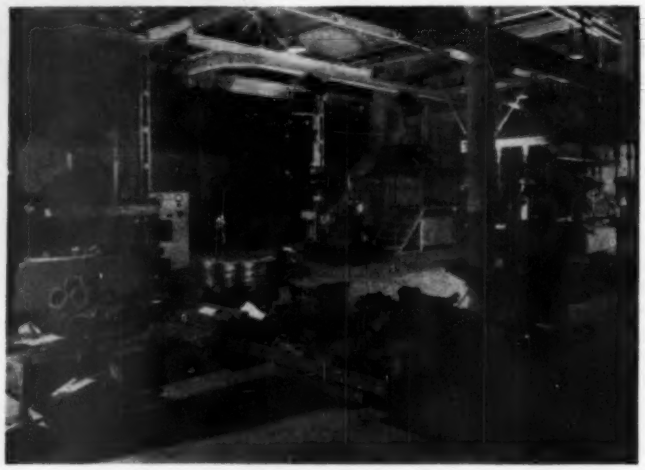
In Spain, as elsewhere, there is an increasing demand for fine steels—today, approximately 7–10% of the total—which, however, presents no problem, as the Spanish plants for special steels already have, or could rapidly prepare, a capacity very much in excess of previous years when production was limited by the shortage of

ferro-alloys, the importing of which is now liberalised. A considerable advance in the production of flat products is forecast which is expected to reach 40% of the total rolled. This tendency corresponds with the substitution of cast steel in many applications; with the more general employment of welding which has considerably modified constructional design; with the use of special profiles obtained from flat products; and with the increase in the manufacture of vehicles and domestic electrical articles.

Increasing availability of supplies and the liberalisation of trade will have a considerable influence upon the small incomplete cycle installations which have been put down in Spain in times of acute shortage, necessitating radical changes in policy and definite specialisation.

Preparation for Painting

New Department for Shot-Blasting and Phosphate Treatment Begins Operation at Leyland Motors' Farington Works



In this general view of the new pretreatment department the Wheelabrator is seen on the left, the final rinse bath in the centre, and part of the continuous overhead conveyor on the right.

A NEW department which recently commenced operation at the Farington works of Leyland Motors, Ltd., will provide additional protection from corrosion on practically every bus and truck component. All cast iron and malleable cast iron components passing through the department will be shot-blasted, painted and force dried, whilst steel components such as cross-members, flitches, bumpers, etc., are to be treated by a phosphating process. Situated in an old part of the engine production plant and covering an area of 7,000 sq. ft., the new department has ample stillage space.

Shot-blasting is carried out by a giant 4-table Wheelabrator, in which the abrasive is flung at the iron components by centrifugal force. The components are placed on the revolving circular tables of the machine, the tables themselves being mounted on a revolving base which, when set in motion, carries the components behind a rubber curtain and through the shot chambers, returning full circle to the loading point.

From the Wheelabrator, the iron components are placed on a continuous overhead conveyor, which carries them at 4 ft./min. past a water-wash paint-spray booth, and through a 15 ft. long infra-red stoving oven. They are sprayed with red oxide of iron zinc chromate primer conforming to DEF.1035B, and passed through the oven at a temperature of 450° F.

Phosphating is carried out by the immersion method, making use of four baths arranged in horseshoe shape served by five 10-cwt. electric cranes on an overhead runway. The four baths are gas heated, and each has an effective compartment size of 12 ft. by 3 ft. by 3 ft. Steam is drawn off the tops of the baths by vents alongside and exhausted through a trunking arrangement with a 30 in. diameter multi-vane fan.

The steel components for phosphating are first degreased in a tank which contains trichlorethylene vapour and then placed in a second bath, which contains a phosphating solution conforming to DEF.29. Independent of bulk, the components are immersed for 20 minutes at a solution temperature of 210° F., and are given a rust inhibiting coat of iron phosphate. Next come rinses in the two final baths at a temperature of 180° F. After rinsing and drying, the components are transferred to the paint spray conveyor.

Heavy Duty Roll Grinder

A £41,000 extra heavy duty roll grinding machine has been ordered from Craven Brothers (Manchester), Ltd., by James Booth Aluminium, Ltd., for installation in their Kitts Green Works, Birmingham. The machine will be used—among other purposes—to grind the rolls of the company's new 148 in. four-high hot aluminium rolling mill which is planned to be in operation during 1961. It will accommodate rolls up to 62 in. diameter, 26 ft. long and weighing up to 75 tons, and is designed both for rough grinding and for obtaining a first-class, ripple-free surface finish. A superfine in-feed of 0·0001 in. per revolution of handwheel is provided. For grinding camber rolls—with either convex or concave surfaces—the grinding saddle of the machine is fitted with Craven cambering gear. Roll journals from 18 in. to 40 in. diameter can be accommodated.

The Corrosion Resistance of Cast Nickel-Molybdenum-Iron Alloys

By G. N. Flint, D.F.C., B.Sc., A.R.I.C., A.I.M.*

Under severely corrosive conditions it is sometimes necessary to use constructional materials having good mechanical properties and a high intrinsic corrosion resistance even in the absence of a protective film. Such materials include the nickel-molybdenum and nickel-molybdenum-iron alloys, and in the present article the author reports test results which show the effect of composition and heat treatment on their hardness and resistance to corrosion by strongly acid media.

WITH the growing complexity of chemical processes and the continued change from batch to continuous operation, increasingly severe demands are made on materials employed in the construction of chemical plant. Structural materials are frequently required to withstand the effects not only of corrosive attack but also of abrasion. In many cases the traditional materials employed by the chemical engineer, such, for example, as lead or stainless steel, prove unsuitable, due to inadequate mechanical strength or because of the instability, in the more severe conditions, of surface films which, in less arduous service, would confer protection from corrosion. In these circumstances it becomes necessary to use materials which have good mechanical properties and a high intrinsic corrosion resistance even in the absence of a protective film. Among the materials within this classification are the nickel-molybdenum-iron alloys which, even at high temperatures, are outstanding in their resistance to corrosion by hydrochloric, sulphuric and phosphoric acids. These alloys therefore find extensive use in critical applications such as pump and valve parts, heat exchangers, agitators and thermocouple sheaths. This article reports tests which demonstrate how composition and heat treatment can influence the hardness of cast nickel-molybdenum and nickel-molybendum-iron alloys and their resistance to corrosion by strongly acid media.

Experimental Procedure

The alloys for test were melted in a high-frequency furnace and cast in green-sand moulds as 5 in. × 1½ in. diameter bars, which were subsequently heated at 1,180° C. for 5 hours and water-quenched in order to minimise effects due to differences in rate of cooling from the casting temperature. The compositions of most of the alloys are given in Tables I, II and III, while molybdenum and iron contents of the remainder are indicated in Figs. 1 and 2. A specimen of wrought nickel was included in some of the corrosion tests for reference purposes. The machined test specimens were 1¼ in. diameter × ¼ in. thick, with a central suspension hole of ⅜ in. diameter. Some discs of the alloys containing 20% or more molybdenum were re-heated at 800° C. for a period of 5 hours and air-cooled, and in addition, two 30% molybdenum alloys containing, respectively, 0·76% and 6·2% iron, were re-heated at 700° C. for periods varying up to 300 hours. The hardness of the

* The Mond Nickel Co. Ltd., Development and Research Department, Birmingham.

TABLE I.—RESISTANCE OF 20% MOLYBDENUM-NICKEL-IRON ALLOYS TO CORROSION BY ACID SOLUTIONS

Composition (%)				Corrosion Rate (m.d.d.)					Hardness (D.P.N.)	
				Aerated 10% H_2SO_4 at 50° C. 3-Day Test	Aerated 5% HCl at 50° C. 3-Day Test		Boiling 10% HCl 1-Day Test			
C	Si	Mo	Fe	A	A	B	A	B	A	B
—	0·30	21·2	0·14	280	233	215	183	203	167	152
0·01	0·26	21·3	5·26	273	236	236	212	273	160	173
0·01	0·14	20·8	10·6	278	242	255	303	401	164	165
0·02	0·22	21·3	14·0	282	212	285	378	524	172	158
0·02	0·14	20·5	18·8	289	228	265	430	560	160	146
0·02	0·19	20·0	24·7	291	228	410	727	3,110	159	140
0·02	0·15	20·0	28·5	305	245	454	1,370	7,700	164	148

A—Heat treated for 5 hours at 1,180° C. and water-quenched.
B—Further heat treated for 5 hours at 800° C. and air-cooled.

TABLE II.—RESISTANCE OF 25% MOLYBDENUM-NICKEL-IRON ALLOYS TO CORROSION BY ACID SOLUTIONS

Composition (%)				Corrosion Rate (m.d.d.)					Hardness (D.P.N.)	
				Aerated 10% H_2SO_4 at 50° C. 3-Day Test	Aerated 5% HCl at 50° C. 3-Day Test		Boiling 10% HCl 1-Day Test			
C	Si	Mo	Fe	A	A	B	A	B	A	B
0·01	0·28	25·6	0·53	187	148	—	55	59	203	294
0·01	0·43	26·1	4·95	209	149	—	53	73	203	245
0·01	0·23	26·2	9·83	249	155	—	75	95	198	207
0·02	0·09	24·7	15·0	262	163	—	176	233	180	171
0·01	0·03	24·2	19·8	275	171	—	311	590	194	187
0·02	0·12	24·2	24·8	284	181	—	454	8,500	196	231
0·02	0·03	23·8	29·2	348	207	—	1,280	11,400	218	333

A—Heat treated for 5 hours at 1,180° C. and water-quenched.
B—Further heat treated for 5 hours at 800° C. and air-cooled.

TABLE III.—RESISTANCE OF 30% MOLYBDENUM-NICKEL-IRON ALLOYS TO CORROSION BY ACID SOLUTIONS

Composition (%)				Corrosion Rate (m.d.d.)					Hardness (D.P.N.)	
				Aerated 10% H_2SO_4 at 50° C. 3-Day Test	Aerated 5% HCl at 50° C. 3-Day Test		Boiling 10% HCl 1-Day Test			
C	Si	Mo	Fe	A	A	B	A	B	A	B
0·04	0·38	31·0	0·76	55	32	53	29	48	224	413
0·01	0·25	30·8	5·24	53	34	53	40	42	215	218
0·01	0·22	30·1	9·87	84	38	140	38	79	213	220
0·01	0·37	30·1	14·3	84	63	237	241	775	239	285
0·01	0·27	31·4	20·2	181	158	356	682	1,300	245	310
0·01	0·35	29·4	23·8	193	278	601	1,220	9,410	255	370
0·01	0·25	31·0	27·9	—	320	807	1,790	9,360	301	366

A—Heat treated for 5 hours at 1,180° C. and water-quenched.
B—Further heat treated for 5 hours at 800° C. and air-cooled.

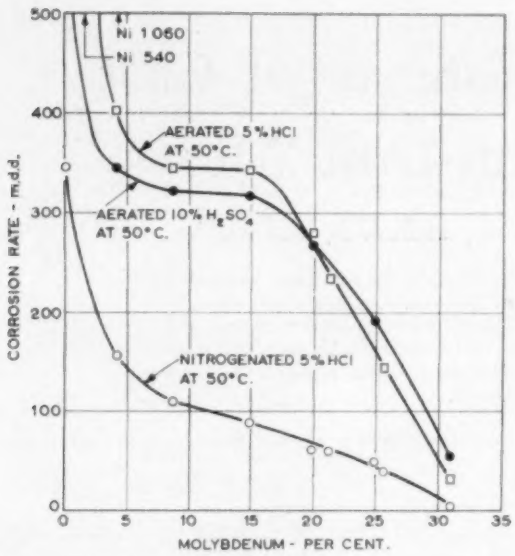


Fig. 1.—Resistance of nickel-molybdenum alloys to corrosion by acids.

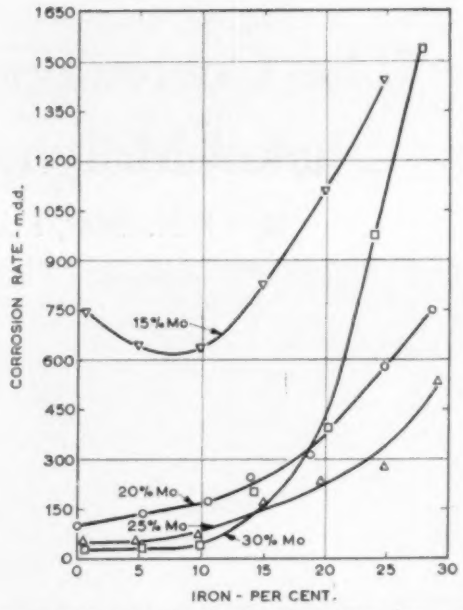


Fig. 2.—Resistance of nickel-molybdenum-iron alloys to corrosion by boiling 5% hydrochloric acid.

low-iron alloy was also determined after treatment at various temperatures between 675° C. and 860° C.

Corrosion tests other than those in boiling liquids were made in a circular-path apparatus, which imparted a motion of 6·25 ft./min. to the specimens. Fourteen specimens were tested in a vessel containing 6 litres of solution, into which air or nitrogen was introduced through sintered-glass diffusers. Tests in boiling acids were made in flasks holding 800 ml. of solution, boiling under reflux: each flask contained two specimens. Rate of corrosion, determined by loss of weight of the specimen, is expressed as milligrams/square decimetre/day (m.d.d.). A micro-examination was made of sections of the corroded alloys.

The carbide present in an alloy containing 30·3% molybdenum and 6·2% iron was extracted by anodic dissolution in a 5% solution of hydrochloric acid in industrial spirits and identified by X-ray diffraction and chemical analysis (see Table IV).

The potentials of the alloys, when corroding freely in unagitated 5% hydrochloric acid at room temperature, were measured relative to a saturated calomel electrode, by means of a valve potentiometer. Anode-polarisation measurements were also made, on nickel and nickel-molybdenum alloys, in an aerated 5% hydrochloric acid solution at 30° C. Soldered connections were made to the specimen discs and insulated by means of a proprietary adhesive resin. The polarising current was supplied from a high-voltage source through a potential divider to a silver cathode of approximately the same surface area as the specimen anode, and was measured on a microammeter. The anode potential determined by means of a valve voltmeter, was measured relative to a saturated calomel electrode.

TABLE IV.—COMPOSITION OF CARBIDE AND OF ALLOY FROM WHICH IT WAS EXTRACTED

Material	Composition (%)				
	C	Si	Fe	Ni	Mo
Alloy	0·03	0·25	6·2	Substantially balance	30·3
Carbide	1·38	3·34	1·8	32·7	58·7

Results of Tests

Influence of Composition on Corrosion Resistance

The results of the corrosion tests on nickel-molybdenum alloys, recorded graphically in Figs. 1 and 3,

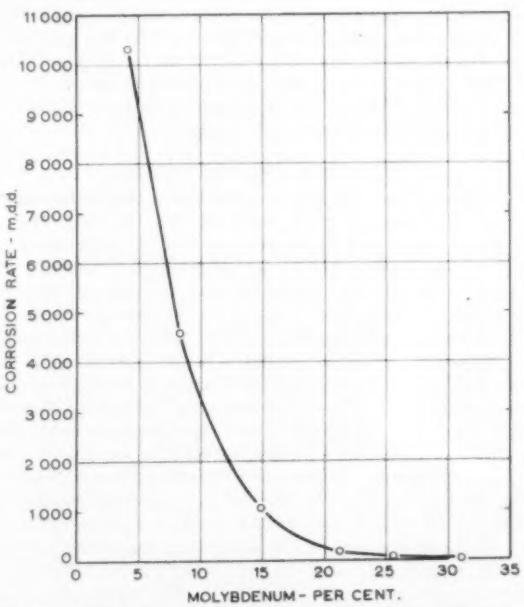


Fig. 3.—Resistance of nickel-molybdenum alloys to corrosion by boiling 10% hydrochloric acid.

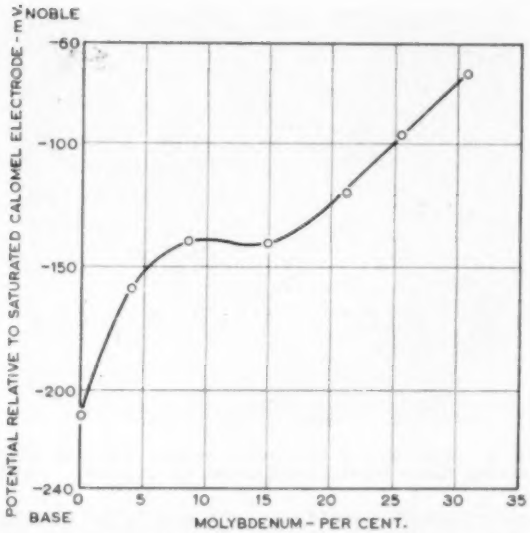


Fig. 4.—Potentials of nickel-molybdenum alloys when corroding in aerated 5% hydrochloric acid at 30° C.

show that the addition of molybdenum to nickel brought about a marked improvement in resistance to corrosion by acid solutions. In the boiling and nitrogenated solutions the extent of the improvement progressively diminished with increase in molybdenum content, but in the aerated solutions there was little difference in the rates of corrosion of the 5, 10 and 15% molybdenum alloys.

Fig. 2, showing corrosion rate against iron content, demonstrates that at all the molybdenum levels studied, iron up to about 10% can be tolerated with little loss of corrosion resistance. With higher iron content, corrosion

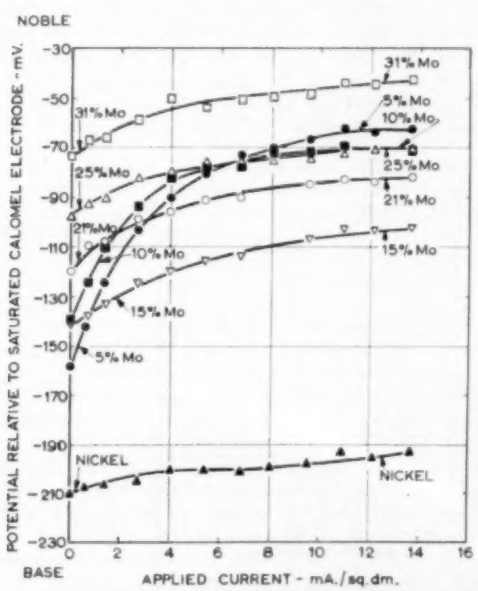


Fig. 6.—Anode-polarisation curves for nickel and nickel-molybdenum alloys in aerated 5% hydrochloric acid at 30° C.

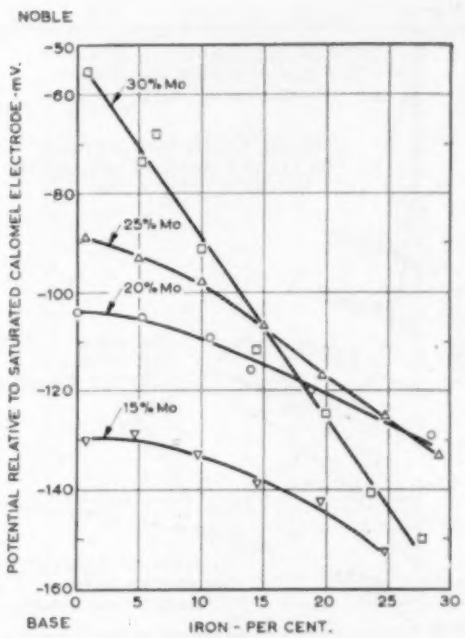


Fig. 5.—Potentials of nickel-molybdenum-iron alloys when corroding in unagitated 5% hydrochloric acid at 24° C.

resistance was lowered—most seriously in the 30% molybdenum alloys. Similar results were obtained in boiling 10% hydrochloric acid, aerated 5% hydrochloric acid solutions and aerated 10% sulphuric acid solution (Tables I, II and III). These results differ from those reported by B. E. Field, [1,2] who found that at the 20% molybdenum level, alloys containing 10% iron corroded at a faster rate than those of higher iron contents, even up to 40% iron.

Potential Measurements

The potentials of nickel-molybdenum alloys corroding freely in aerated 5% hydrochloric acid at 30° C., and of nickel-molybdenum-iron alloys in unagitated 5% hydrochloric acid at 24° C., are shown in Figs. 4 and 5, respectively. With increasing molybdenum content the nickel-molybdenum alloys became appreciably more noble, but there is a step in the curve which shows a striking resemblance to that found in Fig. 1. The addition of iron to nickel-molybdenum alloys caused the potential to become less noble, and the higher the molybdenum content the more marked was this effect.

Anode-polarisation measurements for nickel and nickel-molybdenum alloys are shown in Fig. 6. The outstanding feature of these curves is the high polarisation shown by the 5 and 10% molybdenum alloys; it was appreciably greater than that exhibited by the higher molybdenum-nickel alloys which, in turn, polarised more readily than nickel. The tests were repeated several times, and although the actual potential values showed slight variation, the slopes of the curves remained substantially the same.

On the basis of these results it is possible to suggest an explanation for the fact that in the aerated acid solutions at 50° C. the 10% and 15% molybdenum alloys corroded at about the same rate, whereas in

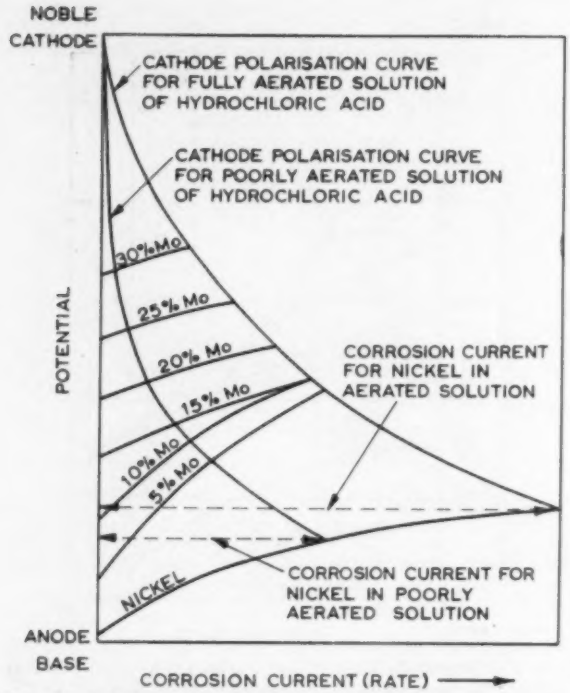


Fig. 7.—Diagram of hypothetical polarisation curves.

unaerated (or boiling) solutions of hydrochloric acid there were usually appreciable differences in the rates of corrosion of these two alloys. If it is assumed that (*a*) the addition of molybdenum to nickel raises the potential of the unpolarised anode by an amount roughly proportional to the amount of molybdenum added, (*b*) in a given corrosive medium the cathode-polarisation curves are the same for all the materials, and (*c*) the anode-polarisation curves are closely similar for aerated and unaerated conditions, it is possible to construct a hypothetical potential/current diagram illustrating corrosion under conditions of full or low aeration. Such a diagram is shown in Fig. 7. The cathode-polarisation curves comprise a summation of the curves for the oxygen-absorption and hydrogen-evolution reactions at the cathode. The curve for the poorly aerated solution, necessarily steeper than that for the aerated solution, cuts the anode-polarisation curves for the 10 and 15% molybdenum alloys before they have opportunity to meet or cross, but in the case of the less steep cathode-polarisation curve for the aerated solution this is not so : hence, the corrosion current intercepts for the 10% and 15% molybdenum alloys show little difference.

The moderately high anode polarisation and nobility of the nickel-molybdenum alloys implies that they will suffer little acceleration of corrosion when in contact with more noble materials such as alloys of nickel-chromium, nickel-chromium-molybdenum and stainless steels when in the passive condition.

Influence of Heat-Treatment on Microstructure, Corrosion-Resistance and Hardness

In the condition obtained by quenching from 1,180° C., the cast nickel-molybdenum alloys containing less than about 15% molybdenum and 0·01% carbon were single-phase in structure, but with increase in molybdenum or

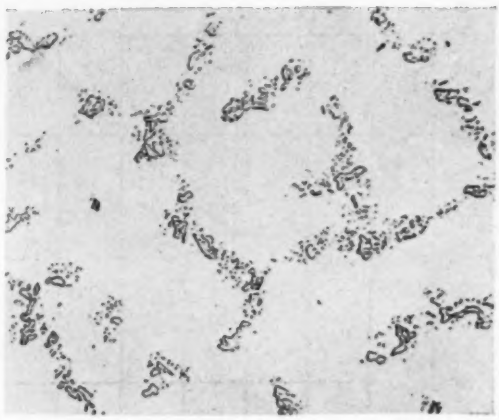


Fig. 8.—Structure of 30% molybdenum, 6% iron alloy, showing interdendritic dispersion of carbide. ×250

carbon contents, carbide was observed in the interdendritic regions. The microstructure of a 30% molybdenum, 6% iron, 0·03% carbon alloy is shown in Fig. 8. Precipitation of carbide was favoured also by increase in silicon or by addition of iron. X-ray and chemical analysis of the extracted carbide showed it to be of the M_6C type, approximating in composition to η_1 (Ni_3Mo_3C), but with insufficient carbon present to satisfy this formula fully : see Table IV. A carbide of similar structure and composition has been found to occur in wrought nickel-molybdenum-iron alloys.[3]

Microexamination of corroded material indicated that the attack occurred preferentially in the regions surrounding the carbide, due, no doubt, not only to the nobility of the carbide but also to the deficiency of molybdenum induced in these regions by precipitation of the molybdenum-rich carbide.

Heat treatment of low-iron 20% molybdenum alloys for 5 hours at 800° C. had no effect on the microstructure, but when the iron content reached 10% a precipitate could be observed in the grain boundaries, and with larger percentages the amount increased, and spread to the interdendritic zones. The precipitate had no effect

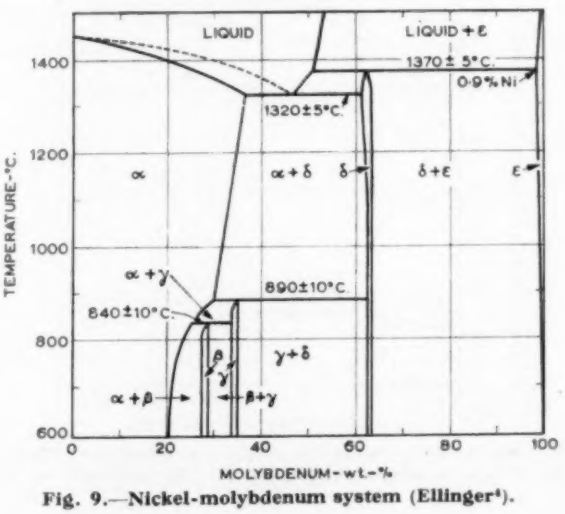


Fig. 9.—Nickel-molybdenum system (Ellinger[4]).

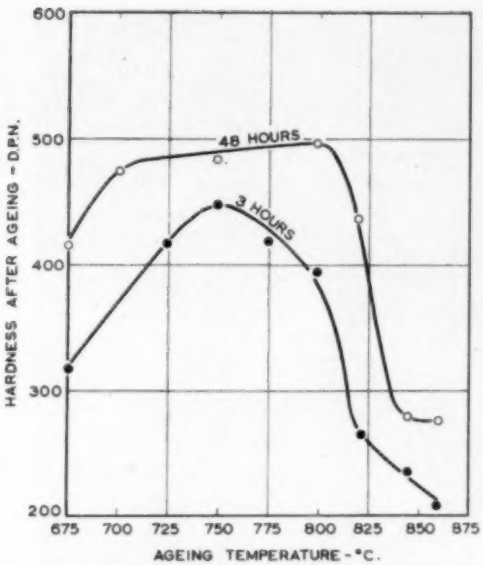


Fig. 10.—Effect of ageing at various temperatures on the hardness of an alloy containing 31% molybdenum and 0·76% iron.

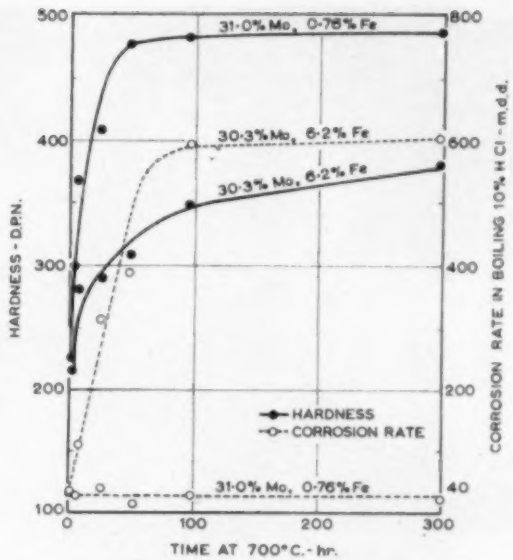


Fig. 11.—Effect of ageing at 700° C. on the hardness and corrosion resistance of 30% molybdenum-nickel-iron alloys.

on hardness (Table I), nor on susceptibility to intergranular corrosion.

At the 25% molybdenum level the 5-hour heat-treatment at 800° C. caused a slight precipitate to form at the grain boundaries which increased in quantity as the iron content of the alloys was raised to 15% (Table II).

The precipitate caused some susceptibility to intergranular corrosion, but in the alloy of lowest iron content this effect was negligible. Alloys containing more than 15% of iron showed precipitation within the grains, associated with deterioration in corrosion resistance. At levels of 25 and 30% iron, precipitation was sufficiently extensive to cause a significant increase in hardness.

The low-iron alloy was appreciably hardened by the heat treatment, and in this connection it is of interest to note, from the nickel-molybdenum equilibrium diagram (Fig. 9),[4] that at 800° C. a 25% molybdenum-nickel alloy is in the $\alpha + \beta$ field and, as will be shown later, it is the precipitation of β which brings about hardening. No increase in hardness was observed in alloys containing 10, 15 and 20% iron suggesting that the addition of iron suppresses precipitation of the β phase.

The heat treatment of alloys containing 30% molybdenum and up to 10% iron for 5 hours at 800° C. resulted in no microstructural changes other than the formation of a fine grain boundary precipitate. Nevertheless, appreciable hardening of the low-iron alloy occurred, but this effect did not extend to the 5 and 10% iron alloys (Table III). In alloys containing iron contents of 15% and upwards extensive precipitation was observed within the grains as well as at the grain boundaries, with the result that hardness was increased and corrosion resistance was markedly impaired.

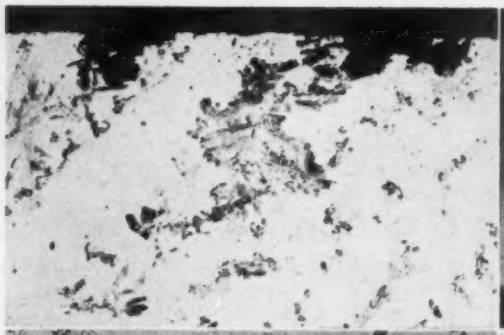
The results of further experiments in which the low-iron alloy was aged at temperatures from 675° C. to 860° C. (Fig. 10) clearly established that hardening was due primarily to the precipitation of β at temperatures below 840° C. and that precipitation of γ, at higher temperatures, had very little influence on this property.

Further attention was given to the two alloys containing, respectively, 31% molybdenum, 0·76% iron, 0·38% silicon, 0·04% carbon, and 30·3% molybdenum, 6·2% iron, 0·25% silicon, 0·03% carbon. These alloys were aged at 700° C. for periods up to 300 hours, and the effect of such treatments on hardness and corrosion resistance is detailed in Fig. 11, which shows that the alloy containing 0·76% iron not only hardened to a greater degree but that its corrosion resistance remained unaffected by the ageing treatment. The effect of iron in impairing the corrosion resistance of nickel-molybdenum alloys and their response to thermal hardening has been noted by Hallet.[5]

The microstructures of the two alloys after ageing for 300 hours at 700° C. are illustrated in Figs. 12, 13, 14 and 15. The low-iron alloy (see Figs. 14 and 15) shows, within the dendrites, striations of the type which have been identified by Ellinger[4] as indicating the presence of β. At the grain boundaries and in certain interdendritic regions, which, due to coring, would be of higher molybdenum content than the dendrites, more extensive precipitation, of a lamellar pattern, may be observed (Fig. 15). From the similarity of these lamellae to those found in photomicrographs shown by Ellinger, it is probable that they consist of the β and γ phases. Despite the grain-boundary precipitation, there was no evidence of intergranular corrosion.

The alloy of higher iron content did not show any striations in the dendrites, but clusters of a lamellar precipitate were observed (see Figs. 12 and 13), and, from their similarity to the structures described by Ellinger[4] and by Stoffel and Stansbury,[6] they were tentatively identified as $\alpha + \gamma$ lamellae. At positions where the clusters outcropped at the surface corrosion was severe (Fig. 12). Fig. 13 also shows faintly, in the matrix, a Widmanstätten precipitate similar to that observed by Stoffel and Stansbury as preceding formation

Fig. 12



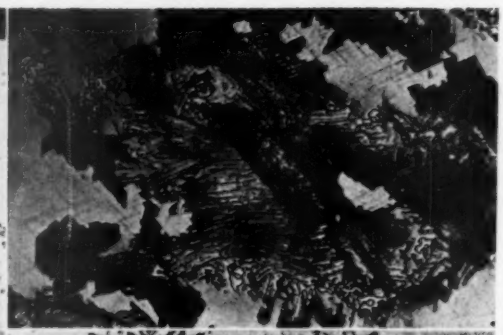
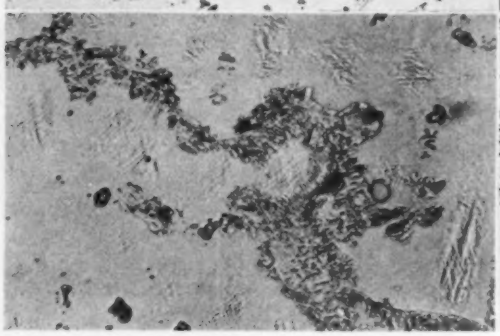


Fig. 13

Fig. 14



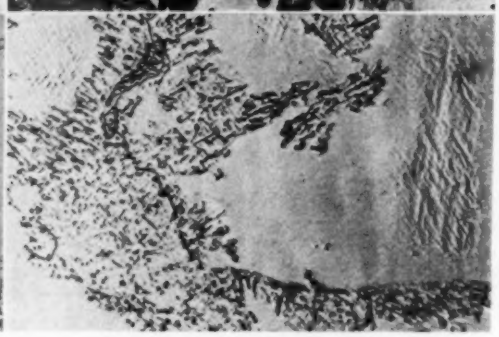


Fig. 15

Fig. 12.—Structure of 30% molybdenum, 6% iron alloy aged at 700° C. for 300 hours, showing localised attack at outcrop of $\alpha + \gamma$ lamellae, after exposure to boiling 10% hydrochloric acid for 8 days. × 100

Fig. 13.—As Fig. 12. × 250

Fig. 14.—Structure of 31% molybdenum, 0·76% iron alloy aged at 700° C. for 300 hours, showing precipitation of $\beta + \gamma$ at the grain boundary. × 250

Fig. 15.—As Fig. 14. × 500

of $\beta + \gamma$ lamellae in nickel-molybdenum alloys containing more than 29% molybdenum.

It may be deduced from these results that the addition of iron to nickel-molybdenum alloys suppresses precipitation of β and favours that of γ; hence it impairs the hardening response of nickel-molybdenum-iron alloys on ageing. Further, since γ is richer in molybdenum than is the matrix, precipitation of γ is more likely to lower corrosion resistance, by molybdenum depletion of the matrix, than is precipitation of β, which has approximately the same molybdenum content as the matrix.

Conclusions

The investigation has shown that for most applications an alloy containing 26–30% molybdenum, 4–7% iron, balance substantially nickel, i.e., the composition of a number of commercial alloys, has optimum corrosion resistance, associated with economy in production, resulting from the use of ferro-molybdenum. Subsequent experience has indicated that the alloy normally has equally satisfactory corrosion resistance in the as-cast condition as after the heat treatment at 1,180° C. for 5 hours employed in the present tests.

For applications which require resistance to both abrasion and corrosion, an alloy containing 30% molybdenum and less than 1% of iron would be preferred, since in the aged condition it has much greater hardness and better corrosion resistance.

Acknowledgments

The author is indebted to The Mond Nickel Co., Ltd., for permission to publish this paper, and to many of his colleagues for helpful discussion.

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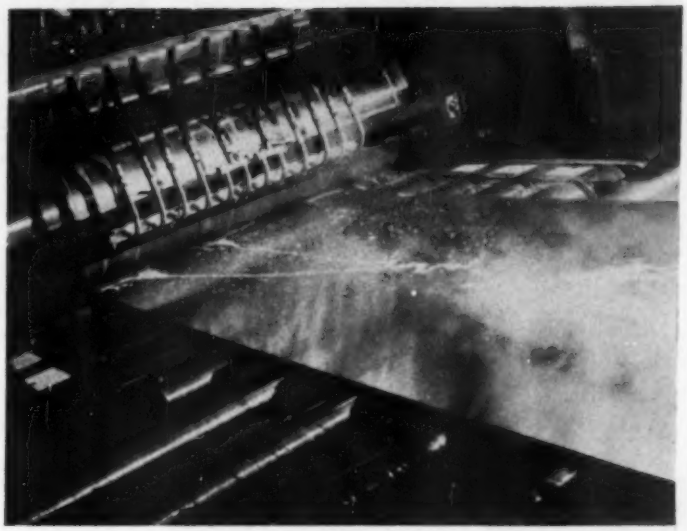
Soviet Journal Translations

A CONTRACT covering the translation into English of twenty-three major Soviet scientific journals, including *Metallurgist*, has been renewed for 1960 and 1961. The original arrangement between Mezhdunarodnaya Kniga (International Book Corporation), and Consultants Bureau Enterprises, Inc., a New York publishing house, was for the year 1958: it was renewed for one year in 1959. Under the terms of the new two-year agreement, the Soviet agency facilitates advance receipt of Russian journals by Consultants Bureau, and is pledged to use all means at its disposal to prevent infringement of the exclusive translation rights by any third party. Original art work and photographs appearing in the Russian periodicals are also rushed to the New York publisher by airmail. In return for these important advantages, Consultants Bureau is making a "reasonable royalty payment." In addition, a special clause in the contract requires that Mezhdunarodnaya Kniga keep the publisher informed of any new scientific and technical journals to be published in the U.S.S.R.

Developments at Alcan's Rogerstone Plant

144 in. Wide Hot Mill is Main Feature



Rolling aluminium plate in the new 144 in. hot mill, the centre-piece of the Rogerstone expansion scheme.

WHEN in September 1950 Alcan Industries, Ltd. (then Northern Aluminium Co., Ltd.) opened its continuous strip mill at Rogerstone—the first of its kind in Europe—it was announced that provision had been made for considerable expansion should market conditions justify it. The ten years that have since elapsed have seen a large increase in the use of aluminium and its alloys—sometimes in directions other than those envisaged at the time. One of the features of this growth has been an increasing demand for plate, which has no doubt been encouraged by the development of easy, reliable welding processes. In the aircraft industry, the adoption of integral construction, in which an assembly of components is replaced by one component machined from plate, calls for plate free from internal defects and from residual stress. To meet these requirements, the company installed new plate handling facilities—commissioned in 1958—which included a 4,000 ton plate stretcher, a 50 ft. ultrasonic inspection tank, a heat treatment furnace, a high speed plate saw, and facilities for the longitudinal welding together of two plates. The necessity for the welding equipment arose from the fact that the shipbuilding industry, which uses large tonnages of aluminium for the superstructures of passenger vessels, required wider plate than could be produced in the existing 96 in. hot mill. This factor, perhaps more than any other, was responsible for the installation of the 144 in. hot mill as the principal feature of the four year expansion programme announced in 1958.

The aim of this development is an increase in output to meet the growing needs of established markets and the provision of means of efficient production of commodities for which a demand has more recently emerged. The scheme concentrates on improvements to the existing continuous strip mill, and covers the installation of new equipment, the modernisation of some existing equipment to increase operating speed and versatility, and at several points the relocation of plant to improve the flow in the mill. When the work is completed the nominal output of sheet and strip will be raised from 50,000 tons a year to 70,000 tons a year, plus a substantial extra capacity for hot-rolled coil and plate. A greater proportion of the output will be of material made from the stronger alloys, and also of aluminium alloy plate, which will be available in substantially greater widths than hitherto.

The expansion has been designed not only to make possible an increased volume of the products for which the mill was originally laid down ten years ago, but also to cater for the demand that has subsequently developed for the stronger alloys and for plate (material $\frac{1}{4}$ in. thick or more). The continuous strip mill was originally designed to handle mainly commercially-pure aluminium (Noral 2S conforming to B.S. 1470: S1C) and the $1\frac{1}{4}$%-manganese alloy Noral 3S (B.S. 1470: NS 3). For the efficient production of the stronger magnesium-bearing alloys in particular, which " work-harden " rapidly during rolling, it was necessary to introduce more powerful, as well as additional, equipment. The result is a much more versatile and flexible production unit, well able to meet the diverse requirements of the increasing number of industries that use aluminium.

Variety of Products

The broadened range of products available from the new mill may be summarised briefly as follows :—

Building Sheet.—This is available for roofing and cladding buildings in a range of profiles in the $1\frac{1}{4}$% manganese alloy, Noral 3S (B.S. 1470: NS3), and is often given a " stucco " finish to reduce glare by rolling between embossed rolls.

Container Sheet.—In the packaging industry, aluminium sheet is competing with tinplate in the manufacture of rigid containers. The gauge ranges from 0·008 in. to 0·018 in., and much of it is in the comparatively hard 2% manganese alloy, Noral M57S (B.S. 1470: NS4).

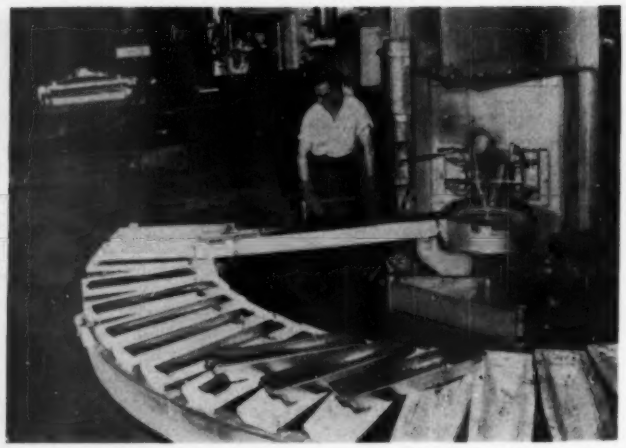
Exterior and chimney of remelt fume-disposal unit.

Foil Stock.—Foil was one of the earliest applications of aluminium in packaging, and in addition to the enormous quantities now used for this purpose, there are substantial outlets for foil in thermal insulation and electric condensers. Alcan Industries do not make foil, but supply the foil producer with annealed coils of high-purity strip for converting into foil as thin as 0·0002 in. without blemish.

Circles.—Circles for the manufacture of domestic holloware are usually supplied in the fully annealed condition, but an interesting Alcan development is the differentially annealed blank, in which the centre of the blank, which will not be subsequently formed, is hard, wheras the edges are soft to permit working.

General Sheet Products.—The broadening of the range of alloys that the mill can efficiently handle means that sheet for innumerable applications will be produced, and in the choice of alloy and temper the producer's experience may be of great value to the user.

Plate.—Reference has already been made to the use of hot-rolled aluminium plate in the aircraft and shipbuilding industry, and as an illustration of the latter, the new ships *Oriana* and *Canberra* embody some 900 tons of 4½% magnesium alloy plate, Noral D54S (B.S. 1477: NP5/6). The ability of aluminium plate to withstand rough usage has led to its use for dumper and tipper vehicles handling rock and ores; for railway mineral wagons; and for coal chutes; whilst its freedom from low-temperature embrittlement makes it the most suitable material for the bulk storage of liquefied gases. The new 144 in. mill brings a big advance in available plate size: from 72 in. to 134 in. in width, and up to 50 ft. long. The high speed plate saw referred to earlier makes possible the supply of plate with edges sawn straight to very close limits.



Casting aluminium pig in the swarf remelt department.

PLANT DETAILS

As mentioned earlier, the expansion at Rogerstone involves the installation of new equipment and the modernisation and reorganisation of existing plant. In the following description, particular attention will be paid to the new items and to the changes made in the original installation, an account of which appeared in *Metallurgia*, 1950, **42**, 223.

Melting and Casting

Aluminium is imported from Canada, where Alcan Industries' associate, Aluminum Company of Canada, extracts it from the ore. It arrives in the form of cast ingots, which after preliminary treatment are ready for rolling, and also as pig which requires remelting and alloying before it can be used in the mill.

At the East Works—another part of the Rogerstone plant—imported pig is remelted and alloyed in large-capacity melting furnaces, the molten metal being poured into water-cooled semi-continuous casting units producing ingots for the mill. Another source of ingot for rolling is the swarf remelt department in which swarf and certain scrap returned from the fabricating processes are recovered by remelting and alloying as necessary.

The swarf remelt department has been moved from its original position to make room for the entry tables of the new 144 in. mill. Of the four furnaces in the original installation only two have been retained. These are of the Birlec low-frequency, vertical-channel induction type, with a melting capacity increased from 7,500 lb. to 10,000 lb., of which 7,400 lb. is now available for take-off. The rating has been changed from 500 kW. single-coil inductors to 200 kW. twin-coil inductors to deal with the increased load.

The melted charge can be syphoned into two holding furnaces, designed by Alcan Industries and heated by nickel-chromium-iron elements suspended in the roof, the maximum load being 165 kW. The capacity of the hearth has been increased from 7,400 lb. to give an off-take of 11,500 lb.

In front of and between the holding furnaces is a modernised semi-continuous casting unit which can be fed from either furnace through a two-branch launder.

This, too, has been subject to modifications involving increasing the maximum drop from 9,000 lb. to 10,000 lb.; introducing controls covering mould cooling and quench water; and installing metal-flow temperature indicators, displayed with a visible ram speed indicator. Provision has also been made to increase the cast length from 120 in. to 150 in. at a later date. A separate high-speed pump for the ram return system has been included and, as before, the unit will cast two ingots simultaneously.

Re-siting this plant has provided an opportunity of installing an improved system of cleaning and removing the fumes that rise both from the furnaces and from the dross that is skimmed off the melt. The former largely arise from the cutting oil on the swarf and scrap, and are dealt with by plant largely designed by the company, with assistance in installation and construction from Thermix Industries, Ltd. The impure air is fan extracted via hoods above the furnaces and washed by water spray, the sludge being clarified and filtered by a Phillips continuously moving paper-belt filter. The high temperature and corrosive nature of the fumes from the dross prevent the use of conventional extraction equipment. Prat-Daniels (Stroud), Ltd., installed the extraction and cleaning equipment which was designed in conjunction with Alcan Industries, Ltd. The stack, internally insulated to overcome the problem of corrosion, is in the form of a venturi and is situated above the dross pot. The fumes are drawn up the stack by means of air driven through a pipe terminating just below the venturi, and are washed at the top of the stack by a water spray, the sludge being treated in a clarifier and a Phillips continuous filtration machine. The cleaned air leaves the stack at 3,000 ft./min.

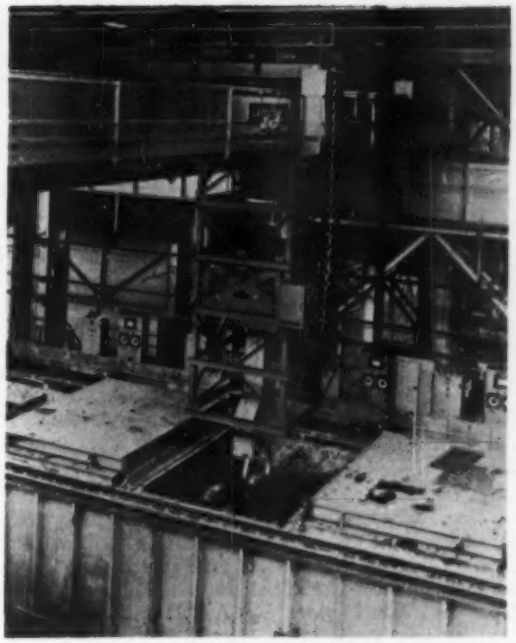
When the new scheme is completed, there will be available an entirely new remelt department, situated on ground reclaimed by diverting the River Ebbw, with facilities for casting ingots up to 8 tons in weight—still supplementary to those imported from Canada. This will form the nucleus of a remelt department that will serve the mill to its ultimate capacity.

Preheating for Rolling

Before preheating, surface roughness and defects are removed in a special scalping (milling) machine which can deal with both faces of a 4,000 lb. ingot in less than 5 minutes. Modifications are in hand to deal with larger ingots and increased production.

After scalping, the ingots are preheated to induce plasticity prior to hot rolling. The four tunnel-type preheating furnaces supplying the existing hot line were unable to deal with the increased size of ingot and volume of production. The required additional capacity has been provided by augmenting the present furnaces by a new bay having four Swindell-Dressler pit-type furnaces, which is designed as the first stage of a preheat department capable of supplying the needs of the continuous strip mill to its ultimate development.

Of the four existing tunnel-type furnaces, two are gas fired and two are electrically heated. The new furnaces, which have been adopted primarily for flexible and economical operation, are indirectly heated by passing air over banks of oil-fired radiant tubes, so that the ingots are protected from the products of combustion. The air is drawn over the radiant tube bank and discharged into



Ingots being placed in pit-type preheat furnace.

plenum chambers at the base of each furnace pit. It then passes up through the load, which is supported by a grill at floor level, and is recirculated. The furnace is constructed of mild steel plate lined with firebrick and hot-face insulating brick, the inside dimensions being 14 ft. wide $\times$ 12 ft. long $\times$ 14 ft. deep.

The operational range of the furnaces is up to 600° C. (preheating temperatures for aluminium alloys range from 480° C. to 600° C.) and the basic performance of a hot furnace is to bring a full load of cold ingots to 600° C. in approximately 8 hours at a heating rate of 17×10^6 B.Th.U./hr. The 2,352 cu. ft. capacity of the furnace can take, for example, a full charge of twenty-two ingots 13 in. $\times$ 48 in. $\times$ 120 in. standing on end and weighing in all 75 tons. The annual capacity of these furnaces is 30,000 finished tons, thus bringing the total ingot preheating capacity to 75,000 finished tons.

The furnace lid is in one piece, constructed of stainless steel panels with glass-wool insulation, and is sealed by asbestos strip. A steel-framed cover lifter of 10 tons capacity, made by Fred Gilbert (Cardiff), Ltd., is mounted on rails on the top of the furnaces. A most important factor in planning the handling operation in this bay was the need to keep to a minimum the time in which the preheating furnaces remain open, and to this end high operating speeds were required. An ingot handling crane, designed and installed by J. H. Carruthers & Co., Ltd., of Glasgow, deals with this problem satisfactorily. The crane is operated by armchair Igranic control from an underslung, insulated and air-conditioned cabin, giving the operator a direct view into the furnace. The main hoist, which is in the form of rotatable tongs with an opening range of 2 ft. 6 in. to 7 ft., has a maximum lift capacity of 8 tons.

It is expected that the preheating capacity will eventually be increased considerably, at which time an

ingot buggy will be provided to deliver the hot ingots to the feed table. Under the present expansion scheme, the preheat and rolling capacity is such that the ingot handling crane can both load the furnaces and transfer the preheated ingots to the feed tables of the mill.

Hot Rolling

The layout for hot rolling consists of a 144 in. four-high mill (which can be used either as the breaking-down mill for the hot line, or on its own for rolling wide plate), a 96 in. two-high mill (which can serve the hot line either as a breaking down mill or as an intermediate roughing mill) and three 88 in. four-high hot-finishing mills working together as a three-stand mill in tandem. On emerging from the last stand, the strip passes through an edge trimmer and immediately to a down-coiler. The original layout in 1950 consisted of the 96 in. mill, followed by an 84 in. two-high roughing mill (which is to be removed) and two 88 in. hot-finishing mills in tandem (to which a third has been added under the new scheme).

All roller tables up to the approach tables of the 96 in. mill have been supplied by Davy and United Engineering Co., Ltd. The 85 ft. length from the preheat bay to the ingoing table of the 144 in. mill comprises thirty-one diabolo-type forged steel rollers in En 8 driven through shafts and bevel gears by A.E.I. mill-type motors.

The main entry table is a turning table and is 24 ft. long and 18 ft. wide; it is formed by fourteen split conical cast iron rollers and each side of the table is driven by a single 75/150 h.p. A.E.I. mill-type motor through line shaft and bevel gearing. Hydraulically operated guides, actuated by a synchronised rack and pinion, are positioned on each side of the table. Once the ingot has been centred, an 8 in. short stroke can be employed which will speed up passes and ensure that when very long slabs are rolled, deviation from the centre on the ingoing side is kept to a minimum. Faces of the guides are fitted with rollers.

The feed table is a separate unit. The four En.8 forged-steel split-conical rollers are each 4½ ft. long (10 ft. wide overall) and are mounted in a frame 14 ft. wide. The rollers are driven through closed spur and idler gears by a 13 h.p. A.E.I. mill-type motor. The table is mounted on an eccentric shaft which is used to adjust the table to the pass line level after roll grinding. It can also be pivoted on the driven roll, which is the outermost, and raised to an angle of 53° by a hydraulic pusher situated in the underframe. When the mill rolls are being changed the table is latched in the raised position to get the strippers on the front of it clear of the roll chocks.

144 in. Hot Mill

The major item installed in the expansion programme is a 38 in. and 60 in. × 144 in. four-high, reversing, hot mill which has been built and installed by Davy and United Engineering Co., Ltd. It is designed to roll ingots 16 in. × 80 in. × 138 in. weighing up to 17,600 lb. The maximum roll opening, however, is 25 in., so that ingots up to that thickness can be rolled in special cases as long as they do not exceed the maximum table loading. The normal duty is either to roll slab 1¼ in. thick for further reduction on the hot line or, alternatively, finished plate down to a minimum thickness of ¼ in. and up to a maximum width from the mill of 134 in. The rolling speed is 298/596 ft./min.

The 144 in. mill is positioned at the head of the hot line in a new bay set at right angles to the original buildings. This bay also houses the new roll grinding equipment and the brick-enclosed 144 in. mill motor room, which is isolated from the main area.

Housings and Screwdown

The 144 in. mill housings, cast by English Steel Corporation, Ltd., each have a finished weight of 163 tons and measure 33 ft. × 15 ft. wide overall. The mean cross-sectional area of each post is 1,420 sq. in.

The screwdown system has two speeds; the faster, 25/50 in./min., is used for normal operations, whilst a speed of ½/1 in./min. is used for screwdown adjustment whilst under load. This second speed will be used in conjunction with a gamma-ray thickness gauge and can at a later date be used with automatic gauge control. Two 200/400 h.p. A.E.I. mill-type motors, mounted on a platform 19 ft. above floor level, drive the screws through a 30 in. Igranic multiple plate clutch and an electrically-operated gear box.

The 20-in. diameter screwdown posts, made of forged steel, have a 1 in. pitch buttress thread fitted in high-tensile bronze boxes set in the housings. The posts act on loadmeters on the top back-up roll chocks, and loads are recorded in the control cabin, from which each screw can be separately operated. A selsyn unit records gap measurement, and limit switches prevent inadvertent over-running.

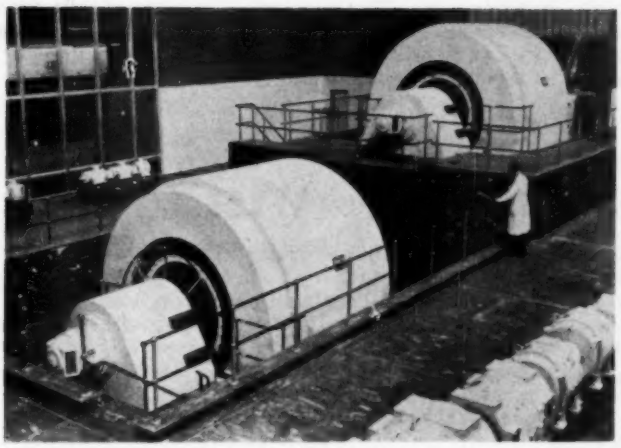
Mill Rolls

The 38 in. diameter work rolls, which each weigh 27 tons, were supplied by William Beardmore & Co., Ltd., and Thomas Firth & John Brown, Ltd. They are made of forged alloy steel, 65°/70° Shore hardness. The minimum diameter to which they may be ground, without affecting the face hardness, is 35⅝ in. The rolls are carried in double-row cylindrical and spherical thrust bearings made by the Skefco Ball Bearing Co., Ltd., and are held in cast steel (Grade B) chocks. The lower work-roll chocks are fitted with hydraulic balancing pushers, operating off the top work roll chocks at 1 ton/sq. in.

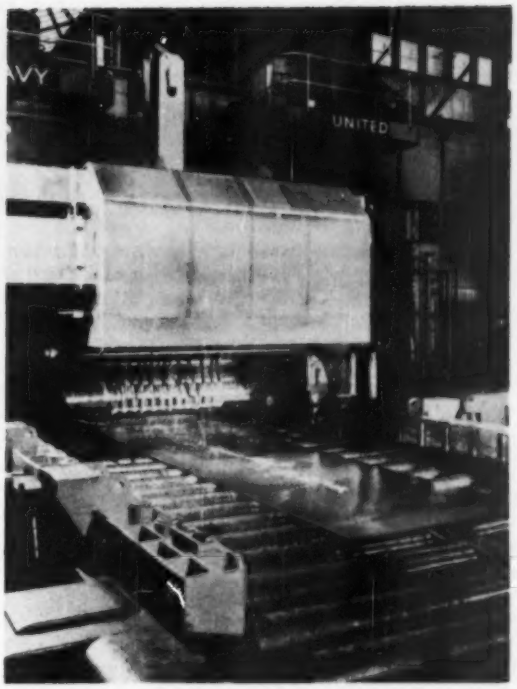
The 60 in. back-up rolls each weigh 68 tons, and were supplied by United Engineering Foundry, Ltd., U.S.A., and by William Beardmore & Co., Ltd. The rolls made by United Engineering are of special alloy, 45°/50° Shore hardness, whilst those supplied by William Beardmore have a composite construction, a forged sleeve (of 50°/55° Shore hardness) on a cast arbor. These rolls may be ground to a minimum diameter of 57 in. without affecting hardness. The roll necks are tapered 1 in 30, and support multi-row Skefco cylindrical and thrust bearings. The top back-up roll chock is latched to a yoke connected by hangers to a forged steel crosshead, which is in turn fixed to a 30 in. diameter balancing cylinder working at 2 tons/sq. in. This cylinder is fitted in the centre of the top housing separator.

Drive

The work rolls are driven at between 30 and 60 r.p.m. (298/596 ft./min. strip speed) by two direct-coupled, 4,000 h.p., single-armature D.C. motors supplied by Associated Electrical Industries, Ltd. A "top forward" arrangement is used in which the top work roll is driven



Main drive motors, each of 4,000 h.p., which are coupled to the work rolls of the 144 in. mill.



View of the 144 in. mill showing manipulator guides, roll cooling sprays and fume exhaust systems.

by the front motor. The rear motor, which is coupled to a jackshaft carried under the front motor, drives the bottom work roll. The arrangement offers easier access for maintenance on the front motor than is possible in the conventional installation of mill drives. The motors are coupled to the work rolls by 39 ft. long forged steel spindles with universal couplings at each end. The top spindle is supported by a carrier bearing mounted in steel beams, pivoted and balanced at the motor end, and hydraulically balanced at the mill end. The lower spindle, between the jack shaft and the bottom work roll, is set in spring balanced carrier bearings at the motor end, and is hydraulically balanced at the mill.

Roll Coolant System

The roll coolant (" soluble oil ") system has a 34,000 gal. main storage tank from which it is supplied by four 1,000 gal./min. centrifugal Pulsometer pumps at 120 lb./sq. in. The Pulsometer pumps are assisted by a 250 gal./min. automatic pressure-booster pump.

The coolant is pumped through a 16 in. Plenty duplex strainer to a battery of three automatic Auto-Klean strainers operating on a differential pressure switch each capable of passing 1,000 gal./min. and filtering particles down to 0·006 in. A Kent combined pressure/flow indicator, with flow recorder, is placed in the line and leads to a British Arca control set, remotely operated from the desk in the 144 in. mill control cabin. The control set can isolate the supply whilst leaving the pumps in operation.

The coolant sprays are operated from the control desk through special Saunders diaphragm control valves, of which 122 are installed at the mill. 6,000 ft. of $\frac{3}{8}$ in. o.d. nylon tube is used between the control desk and the sprays, which permit a maximum flow at the mill of 3,000 gal./min. The coolant, having passed through the sprays, falls to a catch tray under the mill, where it is drained to a 20,000 gal. collecting tank in the oil cellar. Three 15,000 gal./min. 10 ft. head Pulsometer vertical spindle return pumps are provided in this tank for returning the coolant through the cooling tower to the main 34,000 gal. delivery tank in the pumphouse. These pumps are float-controlled, and are fitted with a high-level alarm which can automatically cut-out the main delivery pumps. The booster pump is left running to permit a flow of oil over the mill in case rolling is in operation.

A bank of five electric heaters, which will warm the soluble oil to working temperatures of up to 150° F., is installed in the roll coolant system. This is necessary in order to warm the rolls before rolling, and to help to ensure the rolled aluminium does not cool too quickly. The temperature of the soluble oil can be altered to suit the alloy being processed at the time. A reinforced-concrete cooling tower of 1,500 gal./min. capacity, constructed by the Davenport Engineering Co., has been installed to dissipate excess heat gained when rolling.

Fume Extraction

The fume exhaust system was made and installed by the Visco Engineering Co., Ltd., and has a capacity of 70,000 cu. ft./min. Fabricated aluminium hoods are fitted on either side of the mill stand and are connected by aluminium ducting to a large Aerofoil centrifugal extractor fan made by Keith Blackman, Ltd. This 65 in. diameter fan is driven by a 100 h.p. motor and will absorb 92 b.h.p. at 800 r.p.m., a speed that gives the system a rating of 6 in. water gauge. An oil eliminator is provided to separate and recover the coolant from the exhaust gases. It is then returned to the soluble oil system. The ventilation system in the mill oil cellar has a capacity of 6,000 cu. ft./min., and is capable of providing six air changes per hour.

Roll Change

The roll chock assembly, which is mounted on a sledge, lies on the bottom housing separator. All four rolls, or

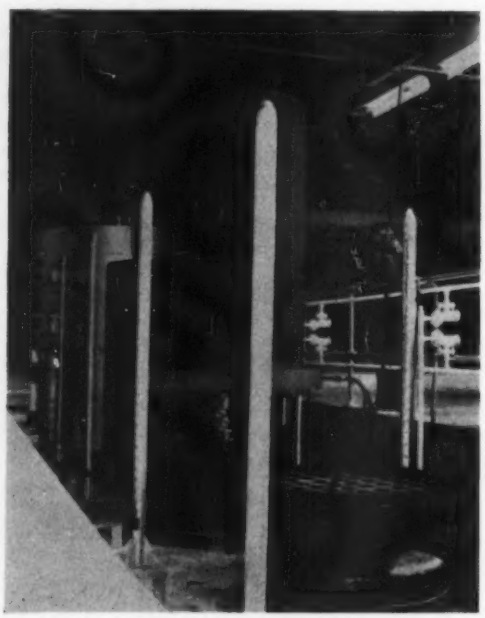
the bottom back-up and two work rolls only, can be withdrawn sideways from the mill on the sledge, which is connected to a rack beam running through a totally enclosed double reduction worm and worm wheel gearbox driven by a 75 h.p. motor. The exposed rolls, with the bearing chocks supported on stools, are removed by a 125 ton E.O.T. crane to the roll-grinding machine, situated in the same bay.

(*to be continued*)

Vacuum Annealing Copper Wire

A BELL furnace installation for the vacuum annealing of copper wire, the first British made installation of its kind, has recently been put into operation in the Hazelhurst Works, of F. D. Sims, Ltd., at Ramsbottom in Lancashire. The plant was designed and supplied by the Electric Resistance Furnace Co., Ltd., and uses an Efco bell furnace, transported by an electric travelling crane to serve six bases where the copper wire, in coil or wound on reels, is heated in vacuum-tight containers. The bases accommodate a stack of wire up to 3 ft. diameter by 4 ft. high, the actual weight of a charge depending upon the size of coils or reels being processed, the maximum weight being about 3,500 lb. The wire is normally heated for 4 hours and then left to cool for from 12 to 20 hours before being exposed to atmosphere. The installation is designed to anneal 34 tons of wire in a working week of 120 hours.

In the short time that has elapsed since the commissioning of the plant excellent results have been achieved. The wire is fully annealed, and it is maintained in a clean and bright condition with complete freedom from sticking between turns. Whilst the plant and its method of operation are similar to many controlled atmosphere bright annealing installations, the use of vacuum is much more economical when compared with the costs of installing and operating a controlled atmosphere generator.



Lowering a self-sealing hood over a charge of copper wire.

The furnace bell is rated at 85 kW. and can operate at temperatures up to 550° C. It has a cylindrical casing of mild steel plate, containing adequate heat insulation with a refractory lining, and being designed to minimise the stressing of the refractory by the repeated raising and setting down of the furnace. The bell is heated by coils of nickel-chromium wire supported around the circumference of the chamber on sillimanite tubes and arranged in three zones, the temperatures of which are independently controlled with automatic indicating instruments. The furnace is accurately positioned over a base by guide posts and the electrical connections are made automatically.

The six bases, which are arranged in a shallow pit to allow sufficient headroom, all have a vacuum-tight steel casing, filled with heat insulation, and carrying on the top a steel charge stand designed to position the charge well into the hot zone of the furnace chamber. The vacuum containers are of plain cylindrical shape with welded, dished tops carrying a single lifting lug. At the bottom of the containers there is a heavy sealing ring having a machined surface which engages with a water-cooled rubber seating surrounding the hearths.

The operating vacuum is obtained with Speedivac pumping gear supplied by Edwards High Vacuum, Ltd. For the initial purging of a container an air-ballasted roughing pump with an air displacement of 15 cu. ft. a minute is used. The process pump has a displacement of 30 cu. ft. a minute. Either pump can be connected through vacuum valves to any one of the bases.

The installation is easily managed by a single operator who stacks the charges on the carriers, places the carriers on the bases below the self-sealing vacuum containers, operates the vacuum pumping equipment and moves the furnace. Throughout the heating period the charges and the furnace bell are protected from overheating by a safety device which gives an aural alarm and removes the supply of the furnace elements.

Mond Labelling Scheme Progress

MANY of the more important out-platers in this country have joined the Mond Labelling Scheme and will supply manufacturers with certified work chromium plated to the requisite grade of the British Standard 1224 : 59. The labelling scheme commenced ten months ago with the object of raising the standard of chromium plating by ensuring that the correct thickness of protective nickel is included in the plating. Out-platers were invited to join the scheme, whereby a label will tell the public that the article has been plated to the British Standard. To date, some fifty of the foremost out-platers are participating so that manufacturers can use the labels, which are supplied at cost by The Mond Nickel Co., Ltd. Goods bearing the labels are now becoming available to the public, enabling purchasers to check that the chromium plating is of high quality.

Investigations on "Active Iron"

Part II.—Techniques Used in Studying Reactivity

By G. V. L. N. Murty, D.Sc., F.R.I.C. and T. R. Ramaswami, M.A., A.R.I.C.

Chemical Laboratories, The Tata Iron and Steel Co., Ltd., Jamshedpur, India.

An apparatus in which active iron, in the form of a deposit on a metal foil, can be made to react with water or a solution of sodium citrate and the evolved gases accurately measured at atmospheric pressure over water has been designed. An elegant and rapid dichromate method for the volumetric estimation of iron in the presence of a large excess of sodium citrate is described.

As indicated in the concluding sections of Part I[1] of this series, systematic investigations regarding the reactivity of the active form of iron involved modifications of the experimental techniques for the measurement of the hydrogen evolved under different conditions and devising new and rapid methods of estimation of iron in the presence of a large excess of sodium citrate.

Measurement of Hydrogen

Active iron, usually obtained as a deposit on a suitable base metal foil, had to be studied as such, due to the rapid oxidation and consequent loss of activity on exposure to air and moisture. The apparatus shown in Fig. 1 was specially designed to handle strips 4×8 cm., the reaction being carried out under water or sodium citrate, and the evolved gases collected over water and measured at atmospheric pressure.

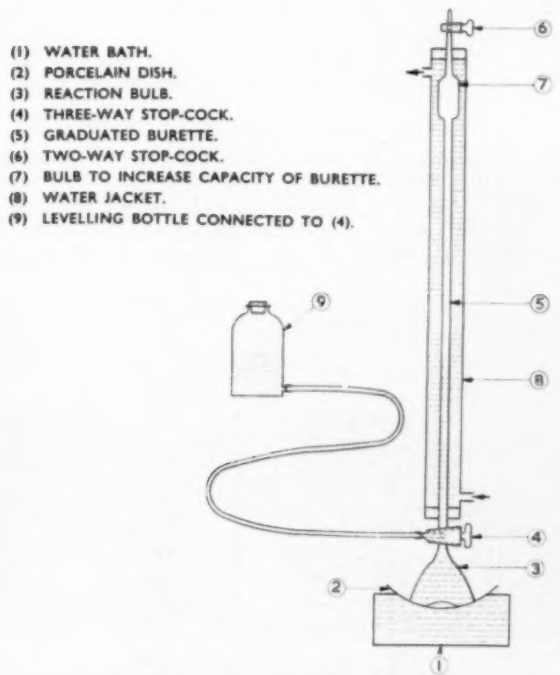


Fig. 1.—Gas measuring burette.

It consists of reaction bulb (3) fitted with a three-way stop cock (4), a graduated burette (5) of 50 ml. capacity carrying a bulb (7) of known volume and a two-way stop-cock (6). The burette and bulb are enclosed in a water jacket (8) to maintain constant temperature. The straight wide bore of the three-way stop-cock (4) connects the reaction bulb and the burette, while the right angle path can be made to connect the burette with the levelling bottle (9) by means of a long rubber tubing. The reaction bulb rests in a 20 cm. diameter porcelain dish (2), and this in turn is placed on a water bath (1). The apparatus is completely filled with water through the levelling bottle by manipulating the stop-cocks (4) and (6) and maintaining a water seal in the porcelain dish (2). If the reaction has to be carried out with sodium citrate, the calculated quantity of this reagent is taken in the dish, instead of water, to give the desired concentration when the bulb is filled completely with water. The strip carrying the active iron deposit is slipped into the reaction bulb quickly, and the volume of the gas that collects in the burette by displacement is measured in the usual way. The solution in the reaction bulb and the gas in the burette can then be analysed.

With the help of this apparatus, the influence of temperature and the area of the deposit on the reactivity of active iron with water and sodium citrate and the mechanism of the iron-water reaction were studied.

Estimation of Iron in the Presence of a Large Excess of Sodium Citrate

During the course of these investigations the need for a rapid and accurate method for the estimation of iron in the presence of a large excess of sodium citrate was keenly felt. The ratio of iron to citrate was often of the order of 1 : 50 and the standard volumetric, gravimetric and colorimetric methods of analysis, involving the preliminary separation or destruction of the citrate, were complicated and tedious.

The recent method based on the photo-chemical reduction worked out by Gopal Rao and Aravamudan[2] did not prove helpful in our ranges of concentration, since even after two hours exposure to sunlight, the reduction of the ferric iron by citric acid was incomplete. However, carrying out the reduction in a Jone's reductor

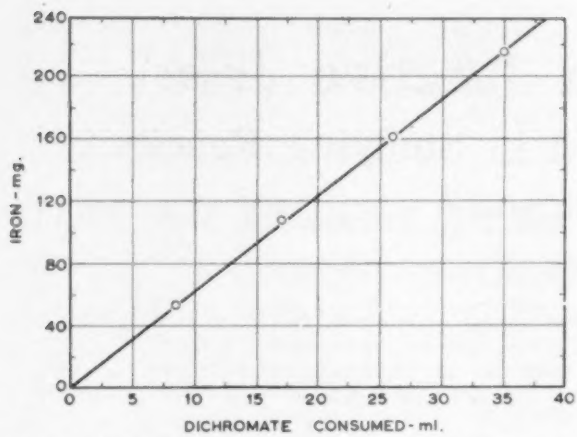


Fig. 2.—Titration of ferrous iron against dichromate in the presence of sodium citrate.

and estimating the iron by titration according to vanadimetric procedure described by Viswanadhan and Gopal Rao[3] yielded satisfactory results. All the same, we felt it desirable to explore the possibilities of working out suitable modifications to the dichromate technique, in view of its ready availability and greater familiarity, in addition to being one of the primary volumetric standards in routine analytical laboratories.

The direct quantitative oxidation of ferrous iron by dichromate in the presence of citrate does not appear to have been attempted in the past, due, obviously, to the ease with which citric acid is known to be oxidised by dichromate in high acid concentrations.[4] But preliminary observations indicated that in an approximately normal sulphuric acid solution, N/10 dichromate showed no oxidising action on citrate at room temperatures (25°–30° C.). However, it was noted that a solution containing ferrous sulphate and sodium citrate consumed more dichromate than could be accounted for by the iron present. This induced oxidation of citrate by dichromate appeared to be influenced by the concentrations of citrate and ferrous iron present in the solution, as well as by the temperature of the reaction. A similar observation was made by Viswanadhan and Gopal Rao[5] with reference to oxalic and citric acids. Systematic work was, therefore, undertaken to standardise conditions for adopting a dichromate procedure. Titrations were carried out with a standard dichromate solution at room temperatures (25°–30° C.) in approximately normal sulphuric acid media after reduction of

TABLE II.—INFLUENCE OF FERROUS IRON CONCENTRATION.

Sample No.	Amount of Iron Present (mg.)	Volume of 15% Sodium Citrate Added (ml.)	Dichromate Consumed (ml.)
1	54	75	8·4
2	108	75	17·0
3	162	75	26·0
4	216	75	34·8
5	324	75	51·5
6	432	75	68·0
7	540	75	83·5

All titrations were conducted in 1N sulphuric acid solution at 25° C.

TABLE I.—INFLUENCE OF CITRATE CONCENTRATION.

Sample No.	Volume of Ferrous Ammonium Sulphate (ml.)	Dichromate Equivalent (ml.)	Volume of 15% Sodium Citrate Added (ml.)	Dichromate Consumed (ml.)
1	20	21·6	1	27·5
2	20	21·6	2	27·8
3	20	21·6	5	30·8
4	20	21·6	10	34·8
5	20	21·6	15	34·9
6	20	21·6	20	34·8
7	20	21·6	75	34·8

All titrations were conducted in 1N sulphuric acid solution at 25° C.

iron by passing through a Jone's reductor. Diphenylamine was employed as internal indicator and the sharpness of the end point improved by the addition of 10 ml. of syrupy phosphoric acid. The titration values were occasionally checked with those obtained by carrying out the titrations using diphenyl carbazide as external indicator. The carbazide was found to be very satisfactory as an external indicator because the colour change was sharp and was in no way vitiated by the presence of zinc in the medium. In this respect, this indicator has a distinct advantage over the conventional ferricyanide solution.

The results presented in Table I clearly show that the volume of dichromate consumed increases with the citrate concentration up to a limit beyond which it remains fairly constant. This important observation was, therefore, harnessed and further investigations carried out which show that, provided the concentration of citrate is maintained sufficiently high, the volume of dichromate consumed is directly proportional to the iron content of the solution. This point is amply illustrated by Table II and Fig. 2. An examination of Table III will show that though temperature exerts a distinct influence on the titration, fairly constant values can be obtained by maintaining it within reasonable limits, viz. 5° C.

Based on the above observations, a satisfactory procedure has been worked out which may be briefly described as follows :—

To a solution containing approximately 200 mg. of iron, 10 ml. of 15% sodium citrate solution is added so that the total citrate content will always be well above

TABLE III.—INFLUENCE OF TEMPERATURE.
(108 mg. iron containing 75 ml. 15% sodium citrate)

Sample No.	Titration Temperature (° C.)	Dichromate Consumed (ml.)
1	10	16·0
2	15	16·4
3	20	16·8
4	25	17·0
5	30	17·2
6	35	17·5
7	40	17·7
8	50	18·0
9	60	18·6*
10	70	19·0*

* End point not permanent.

TABLE IV.—TYPICAL RESULTS.

Sample No.	Iron Content (mg.)	Iron Found by the Present Procedure (mg.)	Iron Found by Conventional Method (mg.)
1	81	84	90
2	108	106	115
3	134	138	145
4	216	218	225

the threshold limit. The resulting solution is rendered nearly 1N with reference to sulphuric acid, after which the entire iron content is brought to the reduced condition by passing through a Jone's reductor. It is then treated with 10 ml. of syrupy phosphoric acid, followed by a few drops of diphenylamine indicator, and titrated against standard dichromate. From the volume of dichromate consumed, the iron content of the solution is directly read off by reference to a standard curve. Care has, however, to be taken to maintain the temperature of titration within $\pm 5^{\circ}$ C. of what had been employed for obtaining the standard curve.

Different solutions containing known and varying quantities of iron and citrate were analysed by the above mentioned method, as well as the conventional volumetric procedure, taking ample care to destroy the citrate followed by a separation of iron by repeated reprecipitation. Some typical results obtained in this regard are incorporated in Table IV. From these it can readily be seen that there is very good agreement between the known quantities of iron taken and the corresponding ones determined by different methods. Thus, the present procedure commends itself for adoption in view of its speed and elegance consistent with accuracy.

Acknowledgments

The authors' thanks are due to Dr. D. Subba Rao for his assistance during the investigations and the management of the Tata Iron and Steel Co., Ltd., for permitting the publication of this paper.

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Water Recovery in Steelworks

Paterson Plant at S.C.O.W.'s Abbey Works

THE Abbey Works of the Steel Company of Wales, one of the world's largest steelworks, with an output of approximately 3 m. tons of steel a year, not unnaturally has a water problem. It became essential to recover as much as possible of the discharged water for re-use in the mill. In modern strip mills the quality of the water must be good, for otherwise rolls will not last and minute indentations will be made on the steel strip which affect the quality of the finished work. Thus, treatment plant for water recovery has to be carefully designed to meet this problem and to ensure that at all times the desired water quality is maintained. The Paterson water treatment plant illustrated successfully treats the effluent from the hot strip mill containing a large quantity of heavy suspended matter and oil, reducing the oil to a negligible quantity and the suspended solids to an average of 14 p.p.m., and on numerous occasions to 5 p.p.m., without filtration.

(Courtesy of The Steel Co. of Wales, Ltd.)

Paterson clarification plant at Abbey Works, showing the Centrifloc clarifiers.

The hot returned effluent is pumped from the scale pits direct to a primary sedimentation tank which is divided into six "canals," each about 100 ft. long by 15 ft. wide, equipped with electrically-driven chain-operated flights of scrapers, the upper flights assisting in the movement of the rising oil to the skimmers and the lower scrapers lifting the heavy suspended solids up to a sloping wall to the top of the primary clarifiers, to be deposited on an endless belt discharging by chute to a railway truck for disposal. The oil skimmers, six in number, are adjustable according to the amount of oil contained in the water and discharge into a holding tank, from whence the oil can be pumped away or removed by mobile tanker.

The effluent then passes through a Venturi flume where it is measured, after which it proceeds to secondary circular clarifiers of the Centrifloc type, each 130 ft. diameter. The Venturi flume is also used to control automatically the coagulants which are added to the water to assist in the precipitation of the lighter and

colloidal impurities. The coagulants are brought by mobile tanker to the chemical house and discharged into holding tanks, from whence they are pumped to the automatic measuring equipment. The coagulants used are waste products and this helps to keep the cost of the treatment to a minimum.

The Centrifloc clarifiers are of the vertical flow type employing a modified sludge blanket with continous sludge discharge. Their design is such as to ensure effective mixing and flocculation of the effluent before it is collected evenly over the upper surface of the clarifiers through radial launders equipped with hydraulically spaced orifices. Each clarifier has a mechanical mixer, mechanical flocculators, and sludge scrapers to remove the deposits formed on the floor of the tank, all the equipment being operated electrically. The design of the Centrifloc clarifier is such that the slurry discharge is kept to a very low figure and is as concentrated as is possible. In these works the slurry is discharged to waste without thickening, but if necessary thickeners can be incorporated and the sludge vacuum dried.

The plant was originally designed for a flow of 18,500 gal./min. but on completion it was found necessary to impose on the plant an overload of about 30%, so that the final flow through the plant is now of the order of 25,000 gal./min.

New Austenitic Manganese Steels

Two new austenitic manganese steels which provide improved abrasion resistance in castings have been developed by Climax Molybdenum Company, a division of American Metal Climax, Inc. One of these steels is a 12-2 manganese-molybdenum alloy which combines toughness and abrasion resistance with a high degree of ductility. The other is a 6-1 manganese-molybdenum alloy which has exceptional abrasion resistance but only moderate ductility.

In both of these steels, molybdenum makes possible the use of higher carbon contents than is normal in Hadfield manganese steels. Where 1·25% carbon often causes embrittlement, the globularising of carbides in grain boundries by molybdenum makes possible the use of as much as 1·7% carbon in commercial castings. Climax has found the new steels particularly valuable in its own mining operations. The 12-2 alloy is being used successfully in slusher scrapers and cone liners. It has demonstrated up to 40% longer life and resistance to flow than Hadfield manganese steel. The 6-1 alloy shows promise for applications such as grinding mill liners and scoop lips, screen decks, grates and jaw crusher liners. The company does not plan to market these steels commercially but will make its research and development information available to interested companies upon request.

Automated Ore Milling Plant

A NEW, highly automated ore milling plant has been opened by The International Nickel Co., of Canada, Ltd., in the Sudbury area of Ontario as the latest step in a continuing programme to counter rising costs with increased production efficiency through technological advances. The modern mill, located at Inco's Levack Mine and built at a cost of 12,000,000 dollars, makes extensive use of instrumentation to permit centralised, and in some cases automatic, control of the crushing, grinding, flotation, and dewatering processes involved in the production of concentrates from ore. All operations are controlled from centrally located instrument panels. Many recently developed techniques, such as the use of radioactive isotopes for making density measurements, have been incorporated into the new plant. The mill, which has a capacity of 6,000 tons of ore per day, is among additions and improvements the company is carrying out at its properties in the Sudbury area which, with the new nickel mining project being developed at Thompson, Manitoba, will raise Inco's nickel production capacity to 385,000,000 pounds per year by 1961. The Levack installation brings to three the number of ore milling plants operated by Inco at its nickel mines in the Sudbury area, the free world's greatest source of nickel.

Meeting Diary

(*continued from page* 182)

8th December

East Midlands Metallurgical Society. "The Fabricating of Steam Generating Equipment for Nuclear Power Stations (with special reference to Welding Problems)," by A. F. GIFFORD. The Faculty of Applied Science, Clifton Boulevard, The University, Nottingham. 7.30 p.m.

Institute of British Foundrymen, London Branch (Beds. and Herts. Section). "Aluminium Castings," by J. VICKERS. Town Hall, Luton. 7.30 p.m.

Institute of Metals, Birmingham Local Section. "Beryllium," by J. WILLIAMS. College of Technology, Gosta Green, Birmingham. 6.30 p.m.

Institute of Metals, Sheffield Section (Joint Meeting with the Sheffield Metallurgical Association). "Dislocations in Metals," by P. B. HIRSCH. Applied Science Building, The University, St. George's Square, Sheffield. 7.30 p.m.

Southamption Metallurgical Society. "Semi-Conductor Materials," by J. G. WILKES. Southampton University. 7.15 p.m.

13th December

Institute of British Foundrymen, London Branch (Slough Section). "Centrifugal Casting of Non-ferrous Metals," by E. AYRES. Lecture Theatre, High Duty Alloys, Ltd., Slough. 7.30 p.m.

Leeds Metallurgical Society. "Metals and Alloys used in the Jewellery and Silverware Industries," by MISS D. PILE. University Staff House, University Road, Leeds. 6.30 p.m.

Sheffield Metallurgical Association, Refractories Group. "Spalling from the First Principles," by H. F. CLEMENTS. 7.30 p.m.

15th December

Powder Metallurgy Joint Group of the Iron and Steel Institute and the Institute of Metals. Discussion of two papers on sintered stainless steel. Church House, Great Smith Street, London, S.W.1. 2.30 p.m.

16th December

Powder Metallurgy Joint Group of the Iron and Steel Institute and the Institute of Metals. "Practical Aspects of Pressing Metal Powders." Church House, Great Smith Street, London, S.W.1.

West of Scotland Iron and Steel Institute. "Recent Developments in Refractories for Iron and Steel Making," by R. MALCOLM. 39, Elmbank Crescent, Glasgow.

21st December

Society of Chemical Industry, Corrosion Group. Open discussion of practical problems. Society of Chemical Industry. 14, Belgrave Square, London, S.W.1. 6 p.m.

An X-Ray Investigation of Systems Between Niobium Pentoxide and Certain Additional Oxides

By H. J. Goldschmidt, D.Sc., F.Inst.P.

The B.S.A. Group Research Centre, Kitts Green, Birmingham, 33

The binary constitutions of niobium pentoxide with the oxides of nickel, cobalt, iron, chromium, vanadium, titanium, zirconium, molybdenum, tungsten, magnesium, calcium, strontium, barium, cerium, aluminium and silicon have been studied on mixtures melted to varying compositions, and in a high and low temperature condition. The primary solid solubilities in β-Nb_2O_5 are frequently extensive and accompanied by a degree of structural distortion. A number of intermediate niobates are formed for higher second-oxide contents and details have been determined; amongst these, the rutile, columbite, hematite, perovskite and other structure types enter dominantly, and their stability fields, transformations, lattice-dimensional and other changes are outlined. A major effect is that of the rutile and columbite-type niobates being in certain cases high and low temperature allotropic forms to one another. Several new compounds and new isomorphs to known structures were found. Homogeneity ranges, which sometimes are considerable and imply varying degrees of non-stoichiometry, are a characteristic of these phases, as well as of the primary end solid solutions. A systematic pattern of phase occurrences emerges and is discussed in the light of the periodic table and crystal chemistry.

IN a recent X-ray high temperature study of allotropic changes occurring in pure Nb_2O_5,[1] the main findings were the existence of two allotropic forms, of which the "high temperature" β modification, supposed stable only above 900° C., was in fact the stable one throughout; whilst the "low temperature" α form was metastable, but could be retained for times increasing with descending temperature before transforming into β. In that paper certain further work on the effect of additional oxides was referred to, and the present account gives a first communication on this.

The questions arise: (1) What would be the influence of foreign oxides, notably those of other transition metals, upon the α- and β-Nb_2O_5 forms (e.g. stabilisation of either, solid solubility, structural distortions); and (2) what new phases and structures would occur at excess concentrations of the second oxide? (The appearance of any such new phases and their equilibria would be of interest in several directions: for instance, in niobium-bearing high-temperature alloys, the existence of such niobates, their physical properties, transformations and volume changes, would have a considerable effect upon the oxidation resistance of an alloy on which they may be present, either in natural scales or protective coatings. Also, some of these compounds would, it is believed, be of interest in the metal ceramics and semiconductor fields.)

Furthermore, a literature search has shown that the systems Nb_2O_5–Me_xO_y (= second metal oxides) are only very incompletely known, so that any basic new knowledge on these systems seemed of some fundamental interest, and this was confirmed as the work proceeded. The existence of several niobium-bearing minerals (e.g. columbite) and synthetic niobates is of course well established, and they will fall into place in the wider pattern emerging.

SYSTEMS STUDIED

In the course of this work on the binary systems between Nb_2O_5 and a second metal oxide, the second oxides being studied include: NiO—Co_3O_4—Fe_2O_3—Cr_2O_3 — V_2O_5 — TiO_2 — ZrO_2 — Ta_2O_5 — MoO_3 — WO_3 — MgO — CaO — SrO — BaO — CeO_2 — SiO_2 — Al_2O_3. (Greater emphasis in study was given to some than to others, as will arise below).

Although many of the metals concerned form, of course, lower oxides than those cited, the principle here adopted is that of using the highest stable oxide to partner Nb_2O_5. It is true that in some cases a pseudo-binary system exists with one of the lower oxides (e.g. with FeO to form columbite $FeO.Nb_2O_5$), whilst for titania addition, for example, the section NbO_2–TiO_2 (both rutiles), as well as Nb_2O_5–TiO_2 becomes relevant. Deviations from stoichiometry clearly play a major part.

EXPERIMENTAL

The two pure binary oxides, in varying molecular ratios were intimately mixed in powder form and compacted, then melted in a laboratory argon-arc furnace under a pressure of 30/40 cm. Hg. The resulting small pellets were examined (1) in the as-melted condition (corresponding, for the rapid cooling rates in the furnace, to the highest temperatures below solidification), and (2) after annealing portions subsequently at 800° C. for 14 days, in order to approach, if not attain, a low-temperature equilibrium in air.

The initial stock of Nb_2O_5 was prepared by oxidising pure niobium metal at about 1,300° C., yielding pure β-Nb_2O_5 (as was verified by X-rays). The final samples were investigated by X-ray powder photography, using established techniques, and structural interpretations and measurement of lattice-dimensions were carried out. A survey of melting points was also undertaken for many of the samples.

The compositions chosen were generally 10, 25, 50 and 75 molecular per cent. second oxide, with, however, additional ones as were required in each case.

It should be mentioned that the compositions here referred to are those "as-weighed," and that the possibility of a minor composition change during melting under argon must be allowed for. The feasibility and success of argon arc-synthesising small experimental ceramic samples was first demonstrated by Geach and Harper[2] and fully supported in the present work.

TABLE I.—SYSTEM Nb_2O_5-NiO

Composition (Molecular % NiO)	Heat Treatment	Constitution	Remarks
10%	As melted	β-Nb_2O_5, very large R (= rutile), very small	
10%	800° C., 14 days	β-Nb_2O_5, large C (= columbite), small	
25%	As melted	β-Nb_2O_5, medium R, large	
25%	800° C., 14 days	β-Nb_2O_5, large/medium C, medium/small	
50%	As melted	R, pure	Spacings : a = 4·717 kX c = 3·038 kX c/a = 0·644
50%	800° C., 14 days	C, virtually pure β-Nb_2O_5, trace	Spacings : a = 4·991 kX b = 12·423 kX c = 5·905 kX
75%	As melted	R, large H (= hematite), medium	
75%	800° C., 14 days	C, medium/large H, medium/large	
90%	As melted	NiO, medium H, medium	
90%	800° C., 14 days	Same + C, small	

RESULTS

Each system will now be dealt with in sequence, with brief specific conclusions, and a more general discussion will follow at the end of the paper. The order will be that of the periodic table. Throughout the tables the rutile-type phases will be designated R, the columbite (niobite)- type phases, C, the hematite-type phases, H, and the perovskite-type phases, P.

System Nb_2O_5-NiO

The constitutions observed in the system Nb_2O_5–NiO are shown in Table I : the following conclusions may be drawn :—

(*a*) Nickel oxide does not stabilise α-Nb_2O_5, but maintains the β-form. The solubility of NiO in Nb_2O_5 is considerable, but somewhat below 10%. It decreases significantly from higher to lower temperatures, so that precipitation (of rutile or columbite, see below) out of a quenched Nb_2O_5 solution is possible

(*b*) A phase of rutile-type structure exists at a composition $NiO.Nb_2O_5$. However, this is stable at high temperatures only, and transforms to one of columbite (niobite)-type structure at lower temperatures. Stoichiometrically the composition of the latter is also $NiO.Nb_2O_5$ (= MeO_2, as rutile), but in fact it appears slightly on the excess NiO side, implying a partial decomposition :

$$\text{Rutile} \longrightarrow \text{Columbite} + \text{trace } Nb_2O_5$$

This non-stoichiometry in nickel columbite tends thus towards excess metal. The important feature, however, is the rutile/columbite transformation as such. This is reversible, in that columbite, once formed at a low temperature, re-forms rutile at high temperatures. Both phases coexist directly with the β-Nb_2O_5 solution.

(*c*) A hematite-type of phase occurs at NiO-richer composition, which itself coexists with rutile at higher, and with columbite at lower temperatures. The formal composition of this phase would be Me_2O_3, but appreciable non-stoichiometry occurs. The hematite phase coexists directly with NiO. No spinel could be detected.

Diagrammatically the position may thus be described in Fig. 1*a**.

System Nb_2O_5-Co_3O_4

The constitutions observed in the system Nb_2O_5–Co_3O_4 are shown in Table II : the following conclusions may be drawn :—

(*a*) Cobalt oxide, like nickel oxide, maintains β-Nb_2O_5 and does not stabilise the α-form. The solubility of Co_3O_4 in β-Nb_2O_5 is considerable, but less than 10% ; it decreases with decreasing temperature.

(*b*) A phase R (rutile structure) exists at an approximate composition $2Nb_2O_5.Co_3O_4$ ($Co_2Nb_4O_{14}$), giving again the formal composition MeO_2 consistent with that correct for rutile and for columbite. Again the columbite structure forms by transformation at lower temperature from high-temperature rutile.

(*c*) On adding further cobalt oxide, the hematite structure is stabilised ; this is at a composition formally consistent with Me_2O_3. The hematite type phase has a small homogeneity range, manifested by a lattice parameter change.

* It should be made clear that these diagrams are based on the two stated heat treatment levels only (i.e. as melted and annealed at 800° C.), but where the constitutions agreed, a joined boundary seemed justified for the purpose of this representation.

TABLE II.—SYSTEM Nb_2O_5-Co_3O_4

Composition (Molecular % Co_3O_4)	Heat Treatment	Constitution	Remarks
10%	As melted	β-Nb_2O_5, large R (= rutile) small	
10%	800° C., 14 days	β-Nb_2O_5, large/medium C (= columbite), small/medium	
25%	As melted	R, large β-Nb_2O_5, small	R—spacings : a = 4·733 kX c = 3·059 kX c/a = 0·646
25%	800° C., 14 days	C, large β-Nb_2O_5, small	C—spacings : a = 5·035 kX b = 14·119 kX c = 5·698 kX
35%	As melted	R, large H (= hematite), medium (C, small)	
35%	800° C., 14 days	C, large H, medium/small	
50%	As melted	H, large R, small/medium	H—spacings : a = 5·155 kX c = 14·095 kX c/a = 2·73
50%	800° C., 14 days	H, large C, small/medium	
62·5%	As melted	H, large CoO, medium/small	
62·5%	800° C., 14 days	Same	
75%	As melted	H, large/medium CoO, medium	H—spacings : a = 5·159 kX c = 14·164 kX
75%	800° C., 14 days	H, large/medium CoO, medium Co_3O_4, small	CoO—spacings : a = 4·24 kX
100%	Original (raw material used)	Co_3O_4 (spinel)* only	
100%	As melted	CoO, large* Co_3O_4, small	CoO : a = 4·26 kX i.e. re-conversion on annealing
100%	800° C., 14 days	Co_3O_4, large CoO, small/medium	

* See text

(*d*) Free cobaltous oxide CoO (NaCl structure) forms the last member in sequence. An anomaly must here be mentioned. The spinel structure of Co_3O_4, although confirmed on the present raw material used, becomes unstable at high temperatures and readily transforms to "CoO," with an oxygen excess lattice (metal vacancies). This was substantiated on melting pure Co_3O_4 in the arc-furnace and in air, which yielded the NaCl type structure of CoO and could be re-converted to spinel by subsequent anneal, though sluggishly. The labile relationship and ready conversion, "CoO" ⟷ spinel is well known; CoO must here be assumed to have absorbed oxygen either by chemisorption or in the lattice. A dissociation temperature $Co_3O_4 \rightarrow CoO$ of 900° C. has been reported. It should be noted that chemically the Co_3O_4 spinel analysed correctly as Co_3O_4, both before and after the conversion to "CoO." Addition of Nb_2O_5 to cobalt oxide maintains the sluggish but reversible $CoO \rightarrow$ spinel change. It contracts the CoO lattice appreciably. This seems unexpected in view of atomic sizes, but would indicate a major metal deficiency developing as some Nb replaces Co.

(*e*) The overall sequence of phases in the Nb_2O_5–Co_3O_4 (CoO) system thus becomes (in terms of type structures) :—

$$\underset{\text{Columbite}}{Nb_2O_5 \rightarrow \underline{\text{Rutile}} \rightarrow \underline{\text{Hematite}} \rightarrow \underset{\text{Spinel}}{\underline{\text{Rocksalt}}}\ (CoO_{1+x}, Co_3O_4)}$$
$$(1) \quad (2) \quad (3) \quad (4)$$

The same sequence of structures, it may be noted, occurs quite generally; in case of the Nb_2O_5–NiO series (see above) it terminates at the rocksalt structure (no spinel); in that of Fe_2O_3 (see below) it terminates at stage (3).

A schematic phase diagram Nb_2O_5–Co_3O_4 is shown in Fig. 1*b*.

TABLE III.—SYSTEM Nb_2O_5–Fe_2O_3

Composition (Molecular % Fe_2O_3)	Heat Treatment	Constitution	Remarks
10%	As melted	β-Nb_2O_5, very large R (= rutile), trace	
10%	800° C., 14 days	β-Nb_2O_5, large C (= columbite), small	
25%	As melted	R, large β-Nb_2O_5, small C, small	R—spacings: $a = 4{\cdot}754$ $c = 3{\cdot}052$ $c/a = 0{\cdot}642$
25%	800° C., 14 days	C, almost pure R, trace	C—spacings: $a = 4{\cdot}756$ kX $b = 13{\cdot}718$ kX $c = 5{\cdot}541$ kX
40%	As melted	R, pure	R—spacings: $a = 4{\cdot}740$ kX $c = 3{\cdot}073$ kX
40%	800° C., 14 days	C, pure	C—spacings: $a = 4{\cdot}948$ kX $b = 14{\cdot}068$ kX $c = 5{\cdot}443$ X
50%	As melted	R, large H (= hematite), small	R—spacings: $a = 4{\cdot}740$ kX $c = 3{\cdot}072$ kX $c/a = 0{\cdot}648$
50%	800° C., 14 days	C, pure	C—spacings: $a = 4{\cdot}973$ kX $b = 14{\cdot}111$ kX $c = 5{\cdot}424$ kX
75%	As melted	H, almost pure R, trace	H—spacings: $a = 5{\cdot}116$ kX $c = 12{\cdot}507$ kX
75%	800° C., 14 days	H, large/medium C, medium	H—spacings: $a = 5{\cdot}212$ kX $c = 14{\cdot}044$ kX
100%	—	H, pure	$a = 5{\cdot}034$ kX $c = 13{\cdot}749$ kX

TABLE IV.—SYSTEM Nb_2O_5–Cr_2O_3

Composition (Molecular % Cr_2O_3)	Heat Treatment	Constitution	Remarks
10%	As melted	β-Nb_2O_5, large R (= rutile), small/medium	
10%	800° C., 14 days	β-Nb_2O_5, large R, small/medium	
25%	As melted	R, large β-Nb_2O_5, small	R—spacings: $a = 4{\cdot}735$ kX $c = 2{\cdot}998$ kX $c/a = 0{\cdot}633$
25%	800° C., 14 days	R, very large β-Nb_2O_5, very small	
50%	As melted	R, pure	R—spacings: $a = 4{\cdot}666$ kX $c = 3{\cdot}003$ kX $c/a = 0{\cdot}644$
50%	800° C., 14 days	R, pure	$a = 4{\cdot}636$ kX $c = 3{\cdot}006$ kX $c/a = 0{\cdot}648$
75%	As melted	H (= hematite), large/medium R, medium	H—spacings: $a = 4{\cdot}948$ kX $c = 13{\cdot}660$ kX
75%	800° C., 14 days	H, large/medium R, medium, but more than as melted	H—spacings: $a = 5{\cdot}233$ kX $c = 13{\cdot}449$ kX
100%	—	H, pure	$a = 4{\cdot}49$ kX $c = 13{\cdot}57$ kX

System Nb_2O_5-Fe_2O_3

The constitutions observed in the system Nb_2O_5–Fe_2O_3 are shown in Table III: the following conclusions may be drawn :—

(*a*) Fe_2O_3 does not stabilise α-Nb_2O_5, but maintains the β-form. A finite solid solubility exists of Fe_2O_3 in Nb_2O_5, but less than 10%; it decreases with decreasing temperature.

(*b*) A phase of rutile structure again occurs, and again transforms to columbite type at lower temperatures. The composition for R is at about 40% Fe_2O_3, but there is a homogeneity range; this stops short of 50%, showing some metal deficiency relative to the MeO_2 composition. The columbite phase C extends over an appreciable homogeneity range, predominating between 25 and 50% Fe_2O_3. This range is reflected by significant changes in spacing.

(*c*) Hematite Fe_2O_3 has a large solubility for Nb_2O_5, decreasing with decreasing temperature under formation of columbite. Considerable lattice spacing changes are caused.

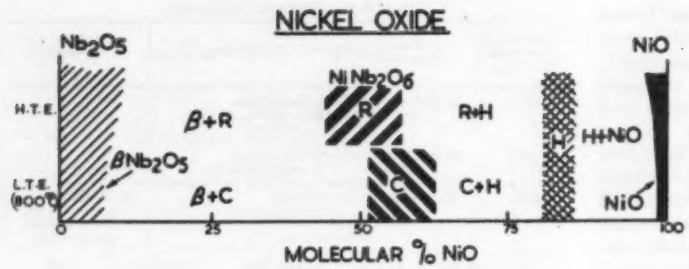
Fig. 1*c* shows the schematic phase diagram.

System Nb_2O_5–Cr_2O_3

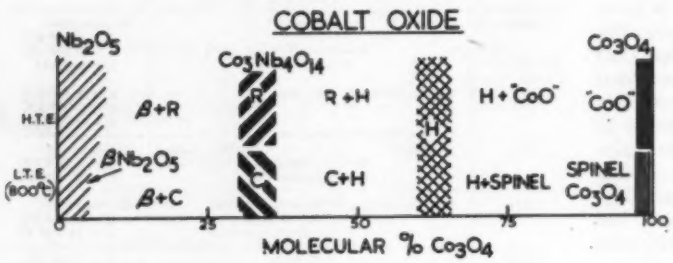
The constitutions observed in the system Nb_2O_5–Cr_2O_3 are shown in Table IV: the following conclusions may be drawn :—

(*a*) Cr_2O_3 dissolves partially in β-Nb_2O_5, but to a considerably smaller extent than nickel, cobalt or iron oxides.

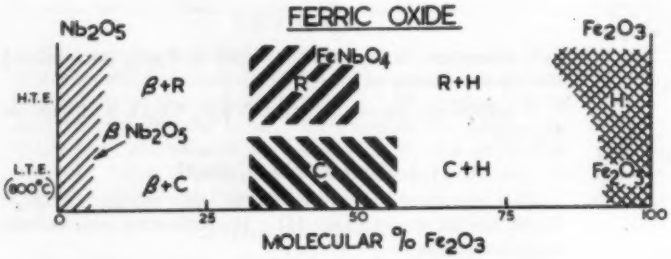
(*b*) The system is dominated by the rutile type structure. This possesses a wide homogeneity range, from the stoichiometric composition $Nb_2O_5.Cr_2O_3$



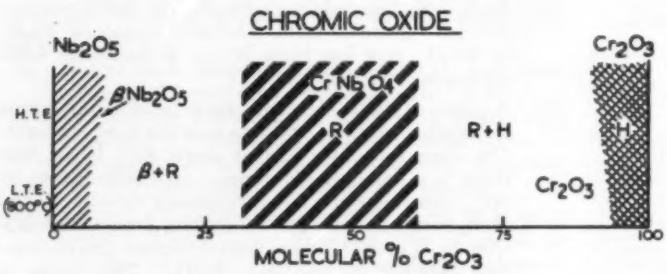
(a) System Nb_2O_5–NiO.



(b) System Nb_2O_5–Co_3O_4.



(c) System Nb_2O_5–Fe_2O_3.



(d) System Nb_2O_5–Cr_2O_3.

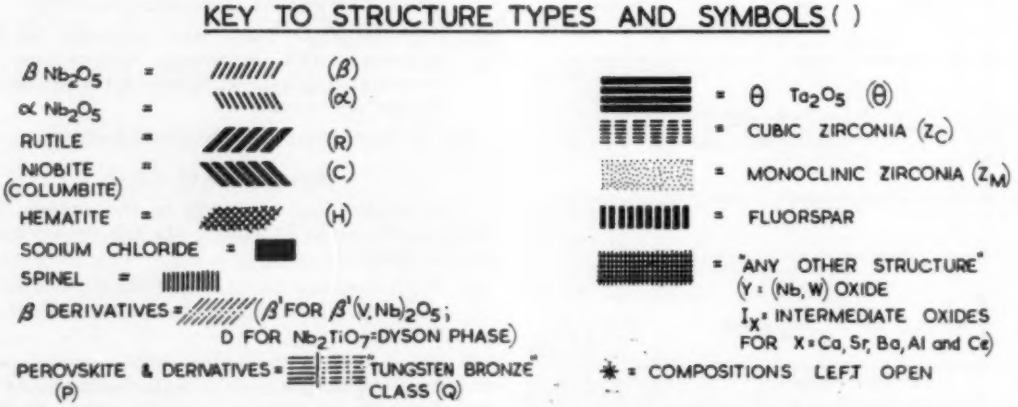
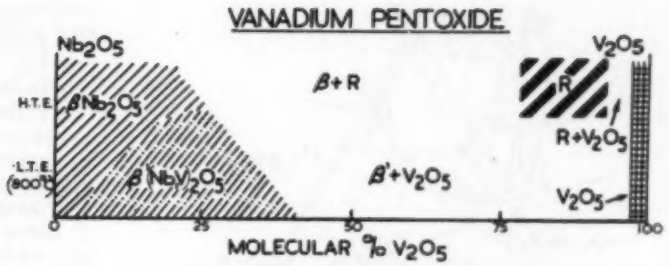
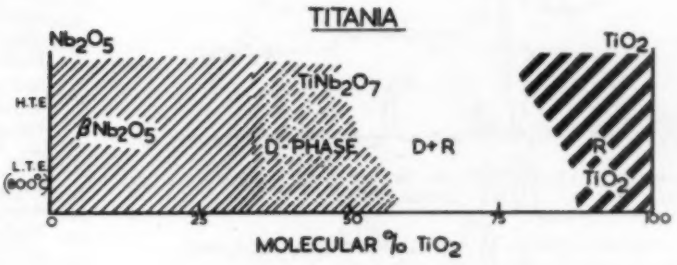

KEY TO STRUCTURE TYPES AND SYMBOLS ( )
βNb_2O_5 = (β)
αNb_2O_5 = (α)
RUTILE = (R)
NIOBITE (COLUMBITE) = (C)
HEMATITE = (H)
SODIUM CHLORIDE =
SPINEL =
β DERIVATIVES = (β' FOR $\beta'(V,Nb)_2O_5$; D FOR Nb_2TiO_7=DYSON PHASE)
PEROVSKITE & DERIVATIVES = (P)
"TUNGSTEN BRONZE" CLASS (Q)
= θ Ta_2O_5 (θ)
= CUBIC ZIRCONIA (Z_C)
= MONOCLINIC ZIRCONIA (Z_M)
= FLUORSPAR
= "ANY OTHER STRUCTURE" (Y = (Nb, W) OXIDE
I_X = INTERMEDIATE OXIDES FOR X = Ca, Sr, Ba, Al and Ce)
= COMPOSITIONS LEFT OPEN

Fig. 1.—Present constitutional results of systems of niobium pentoxide with second oxides.

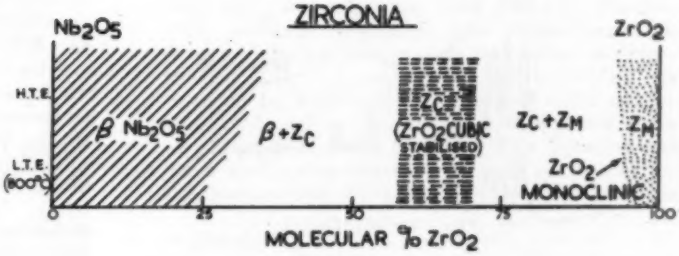
(e) System Nb_2O_5–V_2O_5



(f) System Nb_2O_5–TiO_2.



(g) System Nb_2O_5–ZrO_2.



(= $NbCrO_4$) towards higher Nb_2O_5 contents. The rutile remains stable at both high and low temperatures, and *no* columbite (i.e. no transformation of the rutile) occurs in this system.

TABLE V.—SYSTEM Nb_2O_5–V_2O_5

Composition (Molecular % V_2O_5)	Heat Treatment	Constitution	Remarks
10%	As melted	β-Nb_2O_5, solid solution, pure	
10%	800° C., 14 days	β-(Nb, V)$_2O_5$, pure	β' is close derivative to β-Nb_2O_5
25%	As melted	β-Nb_2O_5, large R (= rutile), small	
25%	800° C., 14 days	β'-(Nb, V)$_2O_5$, pure	
50%	As melted	R, large/medium β-Nb_2O_5, medium	R—spacings: a = 4·624 kX c = 3·015 kX c/a = 0·638
50%	800° C., 14 days	β'-(Nb, V)$_2O_5$, large V_2O_5, small	
75%	As melted	R, large β-Nb_2O_5, small	R—spacings: a = 4·609 kX c = 2·972 kX
75%	800° C., 14 days	β'-(Nb, V)$_2O_5$, large V_2O_5, small/medium	
90%	As melted	R, pure	
90%	800° C., 14 days	β'-(Nb, V)$_2O_5$	V_2O_5 partly vaporised
100%	As melted	V_2O_5	
100%	600° C., 14 days	V_2O_5	No change in structure found

(*c*) The hematite structure of Cr_2O_3 follows next in sequence, dissolving significant amounts of Nb_2O_5: this is accompanied by spacing changes; the hematite solution recedes at lower temperatures, precipitating rutile.

(*d*) The overall phase-sequence in the Nb_2O_5.Cr_2O_3 system thus is :—

$$\beta\text{-}Nb_2O_5 \longrightarrow \text{Rutile} \longrightarrow \text{Hematite}$$

at *all* temperatures, as against :—

$$\beta\text{-}Nb_2O_5 \longrightarrow \underset{\text{Columbite}}{\underline{\text{Rutile}}} \longrightarrow \text{Hematite for } Fe_2O_3$$

A schematic diagram is shown in Fig. 1*d*.

System Nb_2O_5–V_2O_5

The phases observed in the Nb_2O_5–V_2O_5 system are shown in Table V: the following conclusions may be drawn :—

(*a*) V_2O_5 possesses a considerable solid solubility in β-Nb_2O_5 (beyond 10%). This solution transforms however to a phase β', structurally very closely related to β-Nb_2O_5, but with distinctive differences in line intensities and groupings. This is shown in the microdensitometer record, Fig. 2. The β'-phase also possesses an appreciable solubility for V_2O_5; as representative formula $Nb_{1\cdot8}V_{\cdot2}O_5$ may be taken. β' is in direct equilibrium with V_2O_5, but at high temperatures a rutile intervenes, of approximate composition $Nb_{0\cdot2}V_{1\cdot8}O_5$; it decomposes to $\beta' + V_2O_5$.

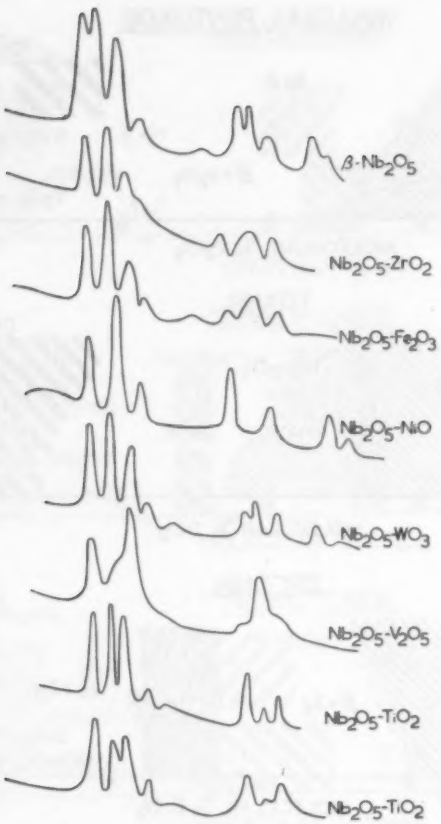


Fig. 2.—Variations in the diffraction pattern of β-Nb_2O_5 as a result of the solid solution of added oxide. (cobalt Kα radiation, spacing range : 4·2> 9> 2·5°A).

(*b*) V_2O_5 itself exists, as far as present observations go, in one crystalline form only, the known one of orthorhombic unit cell.

(*c*) Overall, the equilibria are therefore :

High temperatures : $\beta \longrightarrow$ Rutile $\longrightarrow V_2O_5$
Low temperatures : $\beta \longrightarrow \beta' \longrightarrow V_2O_5$

as indicated in Fig. 1*e*.

Interplanar spacings for the new β' $(Nb, V)_2O_5$ phase are given in Table V*a*.

System Nb_2O_5–TiO_2

The constitutions observed in the system Nb_2O_5–TiO_2 are shown in Table VI : the following conclusions may be drawn :—

(*a*) A wide solubility exists of TiO_2 in β-Nb_2O_5, up to the order of 30%. This is followed by a phase $Nb_2O_5.TiO_2$ agreeing with that reported by Dyson[3] and by Roth and Coughanour.[4] This phase is structurally closely related to β-Nb_2O_5, as shown by only minor differences in powder pattern (see Fig. 2) ; its monoclinic unit cell agrees with that of β-Nb_2O_5 the dimensions being very similar (allowing for double the cell size in β) and as follows :—

βNb_2O_5 : $a = 21·08$kX, $b = 3·82$kX, $c = 19·31$kX, $\beta = 119°42'$
Nb_2TiO_7(D) : $a = 23·8$kX, $b = 3·76$kX, $c = 20·2$kX, $\beta = 120°$

The process, on TiO_2 addition, appears to correspond essentially to a maintenance of the structure, but a limited distortion on passing from β-Nb_2O_5 to Nb_2TiO_7. Already within the β-Nb_2O_5 primary region small modifications in line configuration were observed, corresponding to slight changes preliminary to the "change" to Nb_2TiO_7. Such minor differences occurred not only with the varying composition but also with heat treatment of the same melt. It is doubtful whether Nb_2TiO_7 really ought to be considered a separate phase, because no two-phase field β-Nb_2O_5 + Nb_2TiO_7 occurs, but a continuous structural transition from one to the other. The phase "$3Nb_2O_5.TiO_2$" reported by Roth and Coughanour is viewed here not as an independent one, but as merely a stage in this sequence ; this may however only be a matter of terminology.

(*b*) The rutile structure of TiO_2 is next in sequence, being in direct equilibrium with Nb_2TiO_7. A large solid solubility exists of Nb_2O_5 in TiO_2, extending at high temperature to beyond 20% Nb_2O_5, but receding with lowering temperature so as to give precipitation of Nb_2TiO_7 out of rutile. The rutile lattice is considerably expanded through Nb_2O_5 solution.

A tentative phase diagram, on the basis of the present results is shown in Fig. 1*f*. The position should, however, be viewed in the light of a ternary system TiO_2–NbO_2–Nb_2O_5, because the sub-oxide NbO_2 possesses also the rutile-type structure, and a TiO_2/NbO_2 base solid solution is indeed likely to be stabilised on TiO_2 addition to Nb_2O_5. This would be enhanced in cases of slight oxygen deficiency. Fig. 3 shows a ternary diagram TiO_2–NbO_2–Nb_2O_5, as in principle, it is likely to occur.

System TiO_2–NbO_2

Samples of pure niobium dioxide were readily prepared by melting Nb + Nb_2O_5 mixtures in the correct

TABLE Va.—INTERPLANAR SPACINGS OF PHASE β' $(Nb, V)_2O_5$, SAMPLE : 90% Nb_2O_5, 10% V_2O_5, AS ANNEALED

Interplanar spacing (kX)	Intensity	Interplanar spacing (kX)	Intensity
5·06	M	1·284	M/S
3·76	M/S	1·266	W
3·55	S	1·241	W
3·39	M	1·234	W
3·106	M/S	1·215	M/W
2·894	M	1·191	W
2·713	M	1·159	W
2·495	W/M	1·151	M/W
2·438	W/M	1·139	W
2·331	W	1·117	W
2·147	M/S	1·111	W/M
2·069	M	1·0737	W
1·915	W/M	1·0651	W
1·829	W/M	1·0523	W
1·786	W/M	1·0464	M/W
1·761	M/S	1·0356	M/W
1·738	M	1·0282	M/W
1·682	M	1·0187	M
1·659	M	1·0003	M/S
1·589	W/M	0·9920	M/W
1·564	M	0·9688	M/W
1·558	W	0·9588	M
1·528	W	0·9547	W
1·517	W	0·9473	M
1·474	W	0·9349	M
1·460	M	0·9278	M
1·403	W	0·9226	M
1·320	W	0·9208	M/S
1·301	W	0·9083	M/S

S = Strong. M = Medium. W = Weak

ratio. Using this NbO_2 as one component to a further $50\%NbO_2/50\%TiO_2$ melt, a rutile solid solution was produced; the lattice spacings varied as follows:—

NbO_2:	$a=4\cdot830$kX, $c=2\cdot980$kX, $c/a=0\cdot617$
50/50 NbO_2/TiO_2 (Mol.%):	$a=4\cdot714$kX, $c=2\cdot990$kX, $c/a=0\cdot634$
TiO_2:	$a=4\cdot594$kX, $c=2\cdot958$kX, $c/a=0\cdot644$

Magnéli *et al*[5] had determined the lattice dimensions of NbO_2 as $a=4\cdot836$, $c=2\cdot987$kX, $c/a=0\cdot618$ and shown that the NbO_2-rutile possesses a superlattice, with an actual enlarged unit cell of size $a'=2\sqrt{2}a=13\cdot68$kX, $c'=2c=5\cdot974$kX. The existence of this superlattice is confirmed in the present work, and is already apparent for the $50\%NbO_2$, $50\%TiO_2$ composition, where the actual unit cell size then becomes: $a'=13\cdot33$kX, $c'=5\cdot980$kX.

The overall picture, then, is that TiO_2 permits (1) the solution of Nb in the rutile to give $(Ti, Nb)O_2$, and (2) a change in stoichiometry towards increased oxygen within the rutile lattice. For increased niobium, rutile transforms to the D-phase $TiO_2.Nb_2O_5$ ($=(Ti,Nb)O_{2\cdot3}$), which already conforms to the Nb_2O_5 structure: the stoichiometric stages $MeO_2 \longrightarrow MeO_{2\cdot3} \longrightarrow MeO_{2\cdot5}$ are thus clearly followed, and an intermediate solution series $(Ti, Nb)O_{2\cdot3-2\cdot5}$ exists. (This is believed to be of some basic importance in explaining the favourable oxidation properties of Nb-Ti alloys, and the behaviour of layers of scale on them, containing these oxides.)

System Nb_2O_5–ZrO_2

The constitutions obtained on the Nb_2O_5–ZrO_2 system are shown in Table VII: the following conclusions may be drawn:—

(*a*) A considerable solid solubility exists of zirconia in Nb_2O_5, estimated as in the order of 30% ZrO_2. This again causes a slight modification in, but essentially maintains, the β-Nb_2O_5 structure; a-Nb_2O_5 is not stabilised through ZrO_2.

(*b*) Nb_2O_5 addition to ZrO_2 appears to stabilise the cubic (high temperature) form of zirconia to lower temperatures, so that this phase already occurs at 50% ZrO_2, in equilibrium with β-Nb_2O_5. Further ZrO_2 addition eliminates this phase again, in favour of the low-temperature monoclinic modification of ZrO_2. A compound "$6ZrO_2.Nb_2O_5$," as reported by Roth and Coughanour,[4] could not be confirmed, and was in fact a mixture of the cubic and monoclinic forms of ZrO_2. This question was given special consideration; a melt synthesised to the composition $6ZrO_2.Nb_2O_5$ contained doubtless

TABLE VI.—SYSTEM Nb_2O_5–TiO_2

Composition (Molecular % TiO_2)	Heat Treatment	Constitution	Remarks
5%	As melted	β-Nb_2O_5 solid solution	Slight differences in fine structure and grouping in diffraction lines. Roth and Coughanour's $3Nb_2O_5.TiO_2$ regarded as only one stage, not separate phase. Continuous transition of intensities.
5%	800° C., 14 days	β-Nb_2O_5 solid solution	
10%	As melted	β-Nb_2O_5 solid solution	
10%	800° C., 14 days	β-Nb_2O_5 solid solution	
25%	As melted	β-Nb_2O_5 solid solution	
25%	800° C., 14 days	β-Nb_2O_5 solid solution	
37·5%	As melted	β-Nb_2O_5/" D " Phase	Transitional state
37·5%	800° C., 14 days	β-Nb_2O_5/" D " Phase	
50%	As melted	" D " = $Nb_2O_5.TiO_2$, very large R, small	D = Dyson's phase, $Nb_2O_5.TiO_2$
50%	800° C., 14 days	D, pure	
75%	As melted	R, large D, small	R—spacings: $a=4\cdot704$ kX $c=3\cdot055$ kX
75%	800° C., 14 days	D, large/medium R, medium	
100%	As melted	R	$a=4\cdot594$ kX
100%	800° C., 14 days	R	$c=2\cdot958$ kX

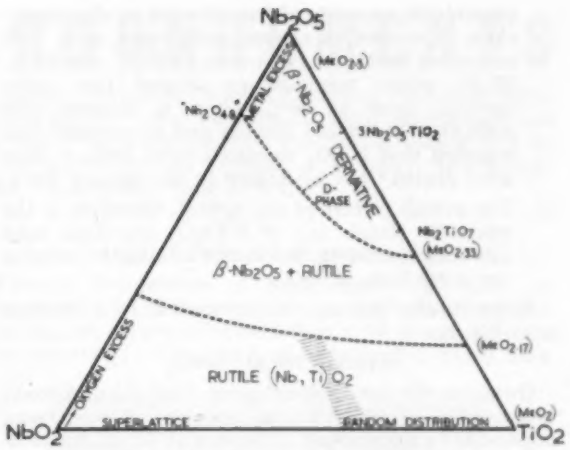


Fig. 3.—Ternary diagram of Nb_2O_5–NbO_2–TiO_2 (illustrative only).

TABLE VII.—SYSTEM Nb_2O_5–ZrO_2

Composition (Molecular % ZrO_2)	Heat Treatment	Constitution	Remarks
10%	As melted	β-Nb_2O_5 solid solution	Slight changes in fine structure of lines
10%	800° C., 14 days	β-Nb_2O_5 solid solution	
25%	As melted	β-Nb_2O_5 solid solution pure	
25%	800° C., 14 days	β-Nb_2O_5, very large Z_c, trace	
50%	As melted	β-Nb_2O_5, large/medium Z_c, large/medium	Z_c = cubic form of ZrO_2 (solid solution)
50%	800° C., 14 days	Same	
62·5%	As melted	Z_c, medium ZrO_2 (mon.), medium β-Nb_2O_5, small/medium	Three-phase coexistences, as pure Z_c unquenchable
62·5%	800° C., 14 days	Same, except less ZrO_2 (mon.)	ZrO_2 (mon.) = monoclinic form
75%	As melted	Z_c, medium/large ZrO_2 (mon.), medium β-Nb_2O_5, small	
75%	800° C., 14 days	Z_c, medium/large ZrO_2 (mon.), medium	
86%	As melted	ZrO_2 (mon.), large/medium Z_c, medium	Composition of $6ZrO_2.Nb_2O_5$, claimed[4] to represent new compound, but not confirmed
86%	800° C., 14 days	Z_c, large ZrO_2 (mon.), small/medium	Cubic ZrO_2 lattices $a_0=5\cdot072$ kX
100%	As melted	ZrO_2 (mon.), pure	A cubic ZrO_2 standard has been prepared by MgO stabilisation

appreciable amounts of free zirconia in the monoclinic form, whilst all remaining lines were well accounted for by the cubic form of ZrO_2. Similarly, Nb_2O_5 richer compositions showed free cubic zirconia, even at 50%, where it coexists with β-Nb_2O_5. Previously Durbin and co-workers[6] had reported that Nb_2O_5 stabilised cubic ZrO_2, a view later denied,[4] but reaffirmed in the present work.

(c) The overall picture of the system, therefore, is the relatively simple one of β-Nb_2O_5 and ZrO_2 solid solutions coexisting, the former tending to stabilise the cubic form of ZrO_2.

Schematically this may be represented by a diagram as in Fig. 1*g*.

System Nb_2O_5–MoO_3

Owing to the low melting point (795° C.) and ready vaporisation of molybdenum trioxide, it was found impossible to bring about reaction with Nb_2O_5 by melting, without substantial volatilisation of MoO_3. The solid remainder proved to be purely β-Nb_2O_5 solid solution; however, modifications in the line-pattern of Nb_2O_5 showed that at least some molybdenum had entered into solution.

The system was not pursued at present, but it may here be reported, as a parallel observation in a separate investigation, that when a series of Nb-Mo binary alloys was allowed to oxidise in air at 1,000° C., the constitution of the scale formed was again purely β-Nb_2O_5 throughout, containing an amount of molybdenum oxide in solid solution (which from line-intensities is apparently constant); this applied to alloys up to at least 90% Mo; the bulk of MoO_3 had evaporated. It seems likely that stoichiometrically the actual lattice solution will have tended towards MoO_2 from $(Nb, Mo)O_{2\cdot5}$, rather than MoO_3, though this is an open question.

(to be continued)

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6 Durbin, E. A., and C. G. Harman: Battelle Memorial Inst. Report 791, 1952. Durban, E. A., H. E. Wagner and C. G. Harman: Ibid., 792, 1952.

Correspondence

A New Version of 'Strength of Materials'

The Editor, Metallurgia.

Sir,

You were good enough to publish much of the work that I did on the mechanical properties of materials in the ten years up to 1952, (*see below*), and I might have offered you more but for a prolonged illness during which I lost most of my papers and lost touch.

If you remember, the whole object of my work was to prove my contention that any of these properties could be calculated from the true stress-strain curve of the material, so that, given the proper stress-strain curve of any material, any of its mechanical properties could be calculated. That was quite a considerable claim, of course, and a completely new one: there was a little occasional appreciation but no criticism, in spite of the fact that my efforts were not always beyond criticism by any means.

As I have explained, I have failed to keep in touch and have no idea whether my work has fallen completely flat or not. Would some of your readers break silence and help me to understand what, if anything, has happened as a result of my efforts for so long?

I should like to ask anyone who would be so good as to come forward with comments and criticism, first of all, whether they agree that I have proved by my work to date that the conversions I claim to be possible are in fact possible. Secondly, I should welcome opinions as to whether it appears to be worthwhile to have all the mechanical properties worked out according to my principles. Thirdly, if the answers to the above are in the affirmative, why does nobody offer to assist me with this work?

Yours faithfully,

A. C. Vivian.

High Orchard,
Ralph Allen's Drive,
Bath.

23rd September, 1960.

Canadian Nuclear Fuel for Japan

Canada's first direct export sale of nuclear fuel elements will be made to the Japan Atomic Energy Research Institute by A.M.F. Atomics Canada, Ltd., Port Hope, Ontario. The contract calls for 270 natural uranium fuel elements for use in a 10 MW. heavy water moderated research reactor. In commenting on the contract, Mr. G. I. Staber, A.M.F. Atomics' general manager said, "This is a significant milestone in the development of industrial participation in Canada's atomic energy program. We have been devoting a great deal of effort to help promote world markets for Canadian uranium products because of the country's vast supply of uranium, and we believe that Canada can become a strong competitor with foreign producers of these products."

More Coke for Steelmaking

Woodall-Duckham Construction Co., Ltd., has received orders to build two new coking plants together valued at over £4 million. Colvilles, Ltd., have ordered for their Ravenscraig works two further batteries of coke ovens with complete coal and coke handling systems and a by-products recovery plant, similar to those under construction at Ravenscraig. By the end of 1962 when all six batteries will be in operation, the Ravenscraig works will be processing 1¾ million tons of coal annually.

The contract for the new coking plant for the United Coke and Chemicals Co., Ltd. (a subsidiary of the United Steel Cos., Ltd.), at Orgreave, includes the demolition of a derelict battery of ovens and the building of a new battery of 43 ovens with by-products recovery and benzole plants and a gas boosting plant.

REFERENCES TO DR. VIVIAN'S PAPERS

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"A Logical Approach to Metallurgical Treatment," Metallurgia, **29**, 15-17
"Strength in Technical Parlance," Metallurgia, **30**, 127-129
"The Relationship of 'Rheology' to Strength of Materials," Metallurgia, **30**, 237-240
"The Shape of a Material's Reactions to Force," Metallurgia, **31**, 225-229, 301-307
"A New Version of 'Strength of Materials,'" Metallurgia, **45**, 29-37

NEWS AND ANNOUNCEMENTS

A Symposium on Welding in Shipbuilding

THE Royal Institution of Naval Architects, the Institution of Engineers and Shipbuilders in Scotland, and the North East Coast Institution of Engineers and Shipbuilders have agreed to co-operate with the Institute of Welding in a joint Symposium on Welding in Shipbuilding to be held in London in October/November 1961.

The Joint Committee invites offers of papers on: the structural design of welded hulls; the building of welded ships; shipyard layout for welded shipbuilding; automatic welding in shipbuilding; materials handling; welded joints in thick mild steel; the use of high yield point steels in welded ship construction; aluminium alloys for welded ship construction; non-destructive testing of ship welds; and training and testing of ship welders. All papers accepted by the Joint Organising Committee will be preprinted and subsequently published with the discussion in a volume of proceedings. The MSS (complete with illustrations) will be required by 1st April, 1961. The preferred length of papers is 2,000 to 3,000 words.

Offers of papers, which should include a synopsis of not more than 200 words, should be addressed to The Secretary, Joint Symposium Committee, The Institute of Welding, 54 Princes Gate, Exhibition Road, London S.W.7 and should reach the Institute before the end of November, 1960.

New Workington Sinter Plant

H.R.H. PRINCE HARALD, the 23-year-old Crown Prince of Norway, is to open a new £2½ million sinter plant at Workington Iron and Steel Company on 6th December. The existing sinter plant at Workington, which has been in operation since 1943, has successfully shown the advantages of using sinter and as additional quantities are now needed the decision was taken to build the new plant. Norwegian iron ore is particularly suitable for the acid Bessemer steelmaking process employed at Workington, and it is supplied in the form of a concentrate which is finely powdered and therefore demands a sintering technique. The company already use up to 150,000 tons of these concentrates each year, which will be increased to 400,000 tons when the new plant is in operation. This will represent about 60% of their raw material intake.

School of Welding Technology

SINCE its foundation by the Institute of Welding in 1957, the School of Welding Technology has organised some fifty courses which have been attended by nearly 2,250 engineers, designers, inspectors, supervisors and other senior personnel from firms, nationalised industries and Government departments. Courses have been organised for the coming months on the following subjects: design for welding in general and mechanical engineering (28th November to 2nd December); brazing —technology and design (16th–20th January); design for welding in thermoplastics (30th–31st January); non-destructive weld examination (13th–17th February); welding of atomic energy plant (27th February–3rd March); advanced course for welding engineers (13th–17th March); and resistance welding—technology and design (27th–30th March). Further particulars may be obtained from the School of Welding Technology, 54, Princes Gate, London, S.W.7.

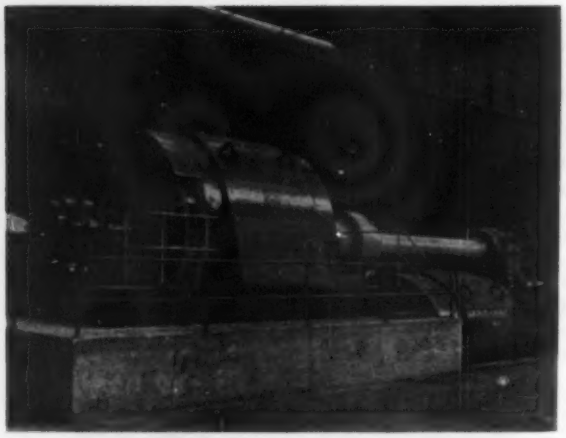
Early Tools of Automation

NEWEST acquisitions of Smithsonian Institution in Washington, U.S.A., are early pyrometers—developed by English emigrant Edward Brown for measuring high temperatures. Examining an 1884 model, which took

much of the guesswork out of steelmaking nearly a century ago, are Mr. R. P. Brown of Philadelphia, the inventor's son (left), and Dr. L. Carmichael, secretary of the museum. On the table is a modern version having automatic control action. The presentation was made by Mr. Brown on behalf of the American associates of Honeywell Controls, Ltd. Mr. Brown and his father helped pioneer the international Honeywell organisation, and the company's instruments, descendants of those designed by Edward Brown, are now widely used throughout the world.

Beryllium Conference

THE Institute of Metals is holding a three-day International Conference on "The Metallurgy of Beryllium" in London on 16th, 17th, and 18th October 1961. The intention is to publish and discuss a great mass of hitherto unpublished original work on beryllium and to cover metal fabrication, physical and mechanical properties, and metal physics—in fact the whole field of the metallurgy of beryllium with the exception of extraction processes. The conference will be organised by the Institute's Nuclear Energy Committee.



Courtesy of Appleby-Frodingham Steel Co.)

The two AEI 3,000 h.p. motors which drive the main rolls of the new 12 ft. reversing plate roughing mill at the Appleby-Frodingham Steel Company's Scunthorpe works: these motors have a peak output of 18,000 h.p.

The organising committee invites offers of original papers for this important conference and requests that prospective authors shall submit firm titles and synopses of their papers by 1st January 1961 and the final manuscripts, in duplicate, not later than 1st April 1961. Prospective authors are asked to advise the Secretary, as soon as possible, of the topics on which they intend to offer papers. Papers will be issued in preprint form in advance of the conference and will later be published promptly, together with reports of the discussions, in bound form. Correspondence regarding the conference should be addressed to the Secretary, The Institute of Metals, 17 Belgrave Square, London, S.W.1.

Coking Delegation in Poland

A DELEGATION of senior officials of the coking industry in Great Britain recently visited research institutions and coke oven plants in Poland. The party, led by Mr. G. W. Lee, Director of the British Coke Research Association, included Mr. D. E. Baird, Director of the British Coking Industry Association, Dr. R. J. Morley, Director-General of Carbonisation, National Coal Board, and Mr. W. J. Pater of the British Coke Research Association. A number of coking plants in Poland were visited and discussions held on technical matters of mutual interest. The tour, which lasted for twelve days, was made at the invitation of the Iron and Steel Board of Poland and was a reciprocal arrangement following the visit of Polish carbonisation officials to Great Britain in December 1959.

Steel Prices

THE Iron and Steel Board has considered the effect on the industry of the recent rises in the price of coal and coke. These have added directly some £13 million a year, or 14s. per ton, to the production cost of steel. After careful consideration the Board have decided not to make any general increase in steel prices as a result of the higher coal and coke prices. On the other hand, this increase in costs has made it impossible to make further steel price reductions in continuation of the downward movement of the past two years. Any further changes which may be made this year in iron and steel prices are likely to be limited to a few products only, or to details of price structure, and are not expected to have any appreciable effect on the general level of steel prices. The Board will continue to keep the question of prices under constant review.

Lecture Courses

Two series of lecture courses have been arranged for the Spring Term of the 1960-61 session by the Division of Metal Science of the Borough Polytechnic, Borough Road, London, S.E.1. Both will comprise six weekly lectures, to be given by specialists, that on recent developments in non-electrolytic metal finishing commencing on 12th January, 1961, and that on the metallurgy of the rarer metals on 7th February, 1961. The topics to be covered by the metal finishing course include the catalytic reduction of nickel from aqueous solutions; galvanising; conversion coatings other than phosphating; phosphating; metal spraying; and hard surfacing. Among the metals to be dealt with in the second course are beryllium, zirconium, tantalum, niobium, tungsten and molybdenum; there will also be lectures on high temperature metals and alloys, and the purification of metals.

Cold Extrusion of Steel Conference

A SPECIAL conference on the cold extrusion of steel, organised by the Institute of Sheet Metal Engineering, will be held in the City Hall, Sheffield from 21st to 23rd November, 1960. Admission to all technical sessions is by ticket only, which are available without charge to members of the Institute and of the National Association of Drop Forgers and Stampers. Non-members may attend on payment of a registration fee of three guineas. Papers to be discussed will deal with various aspects of the production and use of cold extruded and cold forged steel components. Further particulars may be obtained from the Hon. Secretary, Institute of Sheet Metal Engineering, John Adam House, 17/19 John Adam Street, London, W.C.2

Ghana Aluminium Products, Ltd.

GHANA ALUMINIUM PRODUCTS, LTD., announces its intention to install an aluminium rolling mill next to its present factory at Tema. When completed this new industrial project, costing around £1 million, will eliminate the need to import into Ghana many semi-fabricated aluminium products, and it will give employment to over two hundred Ghanaians, many in key positions. The new rolling facilities are expected to be installed and in operation early in 1962. Initially the mill will use imported aluminium ingot, but if the Volta River Project, involving the establishment of an aluminium smelter, goes ahead the mill would be able to use Ghanaian ingot. At present the company is jointly owned by the Government of Ghana and Aluminium, Ltd., of Canada, but arrangements are being finalised for the additional share capital required to finance the rolling mill to be provided by Aluminium, Ltd., the Commonwealth Development Finance Co., Ltd., the United Africa Co. of Ghana, Ltd., Compagnie Francaise de l'Afrique Occidentale, and the Union Trading Co.,

Ltd., with the Government of Ghana retaining its existing investment. The Commonwealth Development Finance Co., Ltd., may also supply part of the additional long term capital which will be needed by the company. Arrangements will be made whereby, when the rolling mill is in full operation, shares in the Company will be available for investment by the Ghanaian public.

United Steel's Output Record

STEEL output of The United Steel Cos., Ltd., was an all-time record at 3,284,170 tons during the financial year ended 30th September, 1960 (53 weeks), exceeding the previous year's output (52 weeks) by 687,298 tons. This is an increase of about 27%. Pig iron production also reached a new high level of 2,008,094 tons, compared with 1,757,413 tons in 1958/59. In addition, new records were established for iron ore and coke production. All the steelmaking branches of the company contributed towards the record results, and as production continues to be maintained at a high level, it is now almost certain that the figures for the calendar year 1960 will show a corresponding improvement over 1959.

Lorries loaded with oil pipeline valves prior to leaving the Penistone works of the David Brown Foundries Division for Liverpool docks. The valves, each of which weighs 6½ tons, will be installed by the Iraq Petroleum Co. in 24 in. diameter pipelines.

Personal News

THE board of directors of Vickers Research, Ltd., is now reconstituted as follows: MR. A. H. HIRD, (chairman), DR. C. F. BAREFORD (managing director), MR. W. D. OPHER, MR. W. D. PUGH, MR. L. REDSHAW, MR. S. P. WOODLEY, M.B.E., MR. R. P. H. YAPP: the secretary is MR. A. D. READ. Research at present being conducted at Sunninghill, near Ascot, on behalf of the Vickers group includes a variety of projects in the sphere of nuclear, medical, chemical, hydraulic and electrical engineering.

MR. J. M. MITCHELL (president of Alcoa International, Inc.) and MR. M. J. S. CLAPHAM (chairman of I.C.I. Metals Division) have been appointed directors of Almin, Ltd. MR. W. BRINING and MR. J. M. GRAHAM (directors of Almin, Ltd.); MR. J. M. MITCHELL; and MR. S. W. WEYSOM (deputy treasurer, I.C.I.) have been appointed directors of Imperial Aluminium Co., Ltd.

IT is announced that for reasons of health MR. T. JOLLY, joint managing director of Guest Keen Iron & Steel Co., Ltd., will retire from his office and from the board on January 1st, 1961.

MR. J. H. GOSS and MR. J. A. RICHARDSON have been elected to the board of the International Nickel Co. of Canada, Ltd. Mr. Goss is vice-president and group executive of General Electric Co., which he joined as a design engineer in 1931, and Mr. Richardson is vice-president of James Richardson & Sons, Ltd., and a director of a number of Canadian companies.

MR. J. BOLTON, secretary for the last six years of the British Steel Castings Research Association, has been appointed director and secretary of the British Steel Founders' Association in succession to MR. R. BARBER, who has resigned in order to take up an appointment in industry. Mr. Bolton is succeeded by MR. E. R. SMITH, who has been in charge of B.S.C.R.A.'s accounts department since 1952. Other B.S.C.R.A. changes include the appointment of MR. C. M. STOCH as assistant director of research. Mr. Stoch has been in charge of the plant engineering and industrial health section since 1953, and for the past two years has been acting head of the steelmaking section. MR. K. KNAGGS, deputy for the past two years, has been appointed head of the steelmaking section. MISS C. M. G. MCBROOM, hitherto private secretary to the director has been appointed technical secretary.

MR. G. H. CLEAVER has been appointed manager of metal research in the Amco division of American Metal Climax, Inc.

THE United Steel Cos., Ltd., announce that following the appointment by Tube Investments, Ltd., of MR. G. E. D. HALAHAN, at present director and general works manager of Steel, Peech and Tozer, as director and general manager of the Round Oak Steelworks, Ltd., from 1st December, 1960, they have appointed MR. H. DARNELL, director and general works manager of Workington Iron and Steel Co. as director and general works manager at Steel, Peech and Tozer. MR. N. D. MACDONALD, at present works manager (iron) at Appleby-Frodingham Steel Co., is to be general works manager of Workington Iron and Steel Co. He is succeeded at Appleby-Frodingham by MR. S. H. BROOKS, with MR. H. S. AYRES as assistant.

MR. G. W. ASHTON, general works manager, and MR. J. ELLIOT, secretary and chief accountant, have been appointed directors of Firth-Vickers Stainless Steels, Ltd.

MR. C. E. PASSMORE has been appointed field sales manager for the Midland region of Griffin & George (Sales), Ltd.

MR. C. E. HOLMSTROM, J.P., chairman of Shepcote Lane Rolling Mills, Ltd., and a director of Firth-Vickers Stainless Steels, Ltd., has retired from the boards of the two companies. MR. J. T. W. DEWAR, managing director of Firth-Vickers Stainless Steels, Ltd., and Shepcote Lane Rolling Mills, Ltd., has been appointed chairman of Shepcote Lane Rolling Mills, Ltd.

Samuel Osborn & Co., Ltd., Sheffield, announce the following appointments to the boards of directors of companies within the group. To the board of Samuel Osborn & Co., Ltd.: Mr. A. Hutchinson, Mr. R. F. Horton, and Mr. I. G. Buchan; becoming local directors of Samuel Osborn & Co., Ltd.: Mr. J. Williams and Mr. V. P. Giles—Mr. F. May to be chairman of the subsidiary board; to the board of Burys & Co., Ltd.: Mr. R. Thompson; to the board of Osborn Foundry & Engineering Co., Ltd.: Mr. A. C. Dennis; to the board of Titanic Steel Co., Ltd.: Mr. F. A. Kirk.

Nash & Thompson, Ltd. announce the appointment of Mr. R. G. Smith as contracts manager, responsible for all sub-contract activities of the company. Mr. A. L. Lee has been appointed sales engineer for the Midlands and Northern England and is responsible for the sales of metallurgical, process control, survey and electronic equipment. Prior to joining Nash & Thompson he was in the technical sales department of Cooke, Troughton & Simms, Ltd., for eight years. Mr. D. H. Yates has joined the company as sales office manager, having previously been with A. H. Hunt (Capacitors), Ltd. He has taken over from Mr. D. Morris who is joining another company. The position of chief inspector has now been filled by Mr. A. Ward who was previously with the Wayne Kerr Laboratories, Ltd., and various Government establishments.

Mr. J. Faulds, formerly a service engineer, then a contracts engineer at the Hillington factory of Kelvin Hughes, has now become the technical sales representative for Kelvin Hughes products in the counties of Northumberland, Durham, Cumberland, Westmorland, North Riding of Yorkshire and Barrow-in-Furness. His address will be 110, Claremont Road, Whitley Bay, Northumberland.

Mr. J. C. Reed, formerly sales engineer of Birmingham Aluminium Casting (1903) Co., Ltd., has been appointed sales manager of the company.

Fletcher Miller, Ltd., manufacturers of cutting and lubricating oils, have appointed as applications engineer at their head office and works, Mr. B. Fraser, formerly assistant chief manufacturing development engineer with the guided weapons division of English Electric Aviation, Stevenage.

West Instrument, Ltd., of Brighton have opened a new sales and service office in South Wales. Situated at 63, The Exchange, Mount Stuart Square, Cardiff, telephone Cardiff 23325, it is to be headed by Mr. L. G. A. Gabe, formerly an executive at the London office of Honeywell Controls, Ltd. Mr. D. R. H. Lane joins the London office staff of the company.

Mr. A. Ratcliffe has been appointed a director of Metals Division, Imperial Chemical Industries, Ltd., and a joint managing delegate director of Marston Excelsior, Ltd., a subsidiary company of I.C.I. At Marston Excelsior, he succeeds Mr. W. Robson who retired on 30th September. Mr. Ratcliffe who will also join the board of another subsidiary, Lightning Fasteners, Ltd., has for the past nine years been production director, and latterly also engineering director, of I.C.I. Salt Division.

Wolf Electric Tools, Ltd., announce the appointment of Mr. D. F. Kershaw as general manager of their Canadian branch.

Obituary

We regret to record the death of the following:

Mr. D. Luther Phillips, who joined the British Iron and Steel Research Association in the early days of 1946, when the Association opened its laboratories at Sketty Hall, near Swansea. For eight years he led the Sketty Hall team in a succession of important research projects related to the coating of steel, when his long connection with local industry was of the greatest value in forwarding the success of the research programme. In 1954 he retired from his position as head of the Sketty Hall Laboratories under age limit, but continued to act in a consultative capacity subsequently.

Luther Phillips' association with the Welsh steel industry was not only of long standing but was very wide. In 1911 he graduated with honours in chemistry at the University College of Wales at Aberystwyth, and was later awarded an industrial bursary at Baldwin's Iron and Steel Works at Landore. Subsequently he became, in succession, chief chemist and metallurgist, and blast furnace manager, and in 1928 was made works manager at Landore. In 1931 he took up the position of senior research officer to the local Steel Tinplate Associations at the Metallurgical Department, University College, Swansea, and in 1937 he visited the United States on behalf of the local Research Committee. It was at that time that his work on strain age hardening culminated in a joint paper with Dr. C. A. Edwards and Mr. H. N. Jones on "The influence of some special elements on the strain age hardening and yield point characteristics of low carbon steels." This paper excited considerable attention as indicating a new and fruitful approach to an important problem.

During his long association with B.I.S.R.A., Luther Phillips made a host of friends, being by nature a man of wide interests and great warmth of character. Everyone who knew him will regret the passing of a delightful personality who carried his learning lightly and earned widespread respect and affection.

Mr. W. H. A. Robertson, founder of W. H. A. Robertson & Co., Ltd., Lynton Works, Bedford, who died at his home in Leighton Buzzard on 21st September at the age of 79. Mr. Robertson was educated at St. Paul's School and at the age of 17 went as a premium pupil to the famous engineering firm Maudslay Sons and Field, Lambeth. After completing his apprenticeship he was employed by the Sturtevant Engineering Co., Ltd., and also W. H. Allen Sons & Co., Ltd., Bedford. In 1904 he joined the Elliott Metal Co. (now part of I.C.I. Metals Division) as chief engineer.

In 1907, at the age of 26, he founded the firm of W. H. A. Robertson & Co., Ltd., now part of the Tube Investment organisation, to manufacture rolling mills and metal working machinery, commencing operations with five employees. Mr. C. E. Davies, now a director of the company and still very active, joined him at the start of the company which now employs about 1,250 people. Mr. Robertson retired from active participation in the management of the business in 1954, but remained a director until his death.

Mr. Robertson was a member of the Institution of Mechanical Engineers, the Institute of Metals, the Iron and Steel Institute and the Junior Institution of Engineers. It was the initiative shown by him that was responsible for the starting of the Institute of Metals of which he was a founder member and later a life member.

RECENT DEVELOPMENTS

MATERIALS : PROCESSES : EQUIPMENT

Gas Welding Accessories

Two kinds of rubber pressure hose designed to give increased safety are now being marketed by British Industrial Gases, Ltd. One is a twin-bore type made of reinforced rubber, each bore having a diameter of $\frac{3}{16}$ in. By splitting the tubes apart at the end of the hose, two connections can be made, while only one length of hose trails between gas cylinders and equipment. Twin hoses with a range of bores of up to $\frac{3}{8}$ in. can also be made available. The other new type of hose is specially designed for use with propane, and is stocked in all sizes. It is lined with nitrile or neoprene, which are not affected by liquid propane and will, therefore, give longer life and increased safety.

New and cheaper oxygen and acetylene regulators (the 69.I and 68.I respectively) in which the cylinder contents are indicated by a calibrated plunger instead of a gauge, have also been introduced recently by the company. The regulators, which are of the single stage type, are designed for rough usage and are small and robust. Special account has been taken of the effect of cylinder movement on regulators, and the new instruments are difficult to damage even with the roughest manhandling of cylinders: they will be of great value in the scrap industry where fine pressure adjustment is unnecessary.

British Industrial Gases Ltd., 700 Great Cambridge Road, Enfield, Middlesex.

Rotary Furnace

Dowson & Mason, Ltd., are now able to make available in this country a gas-fired rotary furnace designed and developed by their American associates, the Rational Furnace Corporation of Providence, R.I. This furnace was designed primarily for heating stainless steel rods prior to rolling to knife-blade thickness in a standard rolling-mill, but it obviously has many other industrial applications.

The furnace body is a welded steel cylinder, with a $7\frac{1}{2}$ in. thick refractory lining, and stands on an angle-iron frame. Inside the furnace chamber is a spider carrying twelve alloy cups, each $4\frac{1}{4}$ in. inside diameter and 10 in. long, equispaced on a P.C.D. of 25 in. The spider, which slopes backwards at an angle of 5° to prevent the contents from falling out of the cups, is mounted on a shaft which protrudes through the back of the furnace chamber to the driving mechanism. The shaft is supported in standard bearings, and heat-losses are minimised by the incorporation of a special heat-seal. The shaft is driven by a $\frac{1}{4}$-h.p. electric motor, and indexed in twelve positions by an air-operated cylinder. The distance between the end of the cups and the front of the furnace may be varied by means of a screw mechanism operated with a handwheel. The front of the furnace chamber is attached to the body with four bolts, and can easily be removed for maintenance.

In the centre of the furnace-front is a gas-burner (with continuous pilot-burner), whose flame plays on a refractory cone cast in the centre of the spider. The angle of the cone is designed to give optimum heat-circulation in the furnace. Interchangeable burners can be supplied for the various uses to which the furnace may be put. In the "three o'clock" position on the furnace front is a square aperture whose centre is on the P.C.D. of the cups, and 42 in. from the ground, which acts both as a loading-hatch, and as a flue. An air-curtain directs the spent gases up a water-cooled chimney.

After the furnace has been brought to working temperature, which takes about two hours from cold, the operator loads the cup behind the hatch with the pieces to be heated. Pressure on a knee-switch causes the spider to turn to the next indexed position, the operator loads the next cup, presses the switch, and so on until all twelve cups are full. By now, the pieces first loaded will have been in the furnace for upwards of two hours, and after the next press of the switch, they may be removed and transferred to the rolls. The distance from the centre of the hatch to the side of the furnace chamber is only 13 in., so that a minimum of heat will be lost in transferring the work. When the cup has been emptied, it is re-loaded before the next cup is brought into position. It will be noted that as the charges are carried round the furnace, each will turn relative to the others, and perfectly uniform heating is thereby ensured.

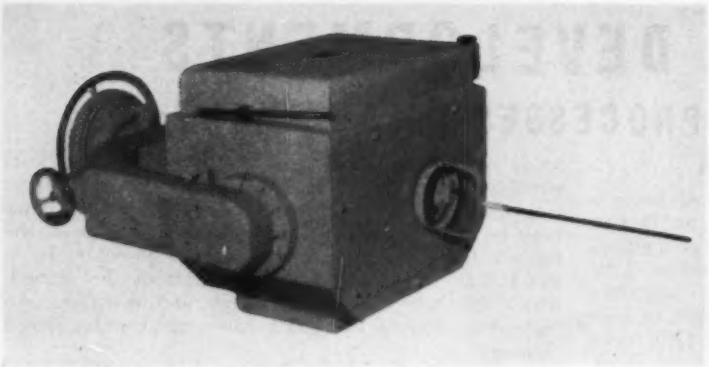
One customer has been using the furnace for heating stainless steel rods $\frac{13}{16}$ in. in diameter and $10\frac{1}{2}$ in. long to a pre-rolling temperature of 1,400° F. (760° C.). Fifty rods are placed in each cup and remain in the furnace for about $2\frac{1}{4}$ hours, giving an output of 250 rods per hour.

Dowson & Mason, Ltd., Alma Works, Levenshulme, Manchester, 19.

15-MeV Mobile Betatron

For non-destructive testing of steel thicknesses of up to 4 in., the majority of supervoltage X-ray units and radioactive isotopes currently used are sufficiently powerful. Above this thickness, however, exposure times become unacceptably long, and the need arises for a powerful X-ray generator which possesses an extremely high degree of sensitivity when dealing with thick structures.

The Siemens 15 MeV mobile betatron, which has been introduced to the market by Pantak Ltd., comprises essentially an electromagnet shaped in the form of a shell-type transformer which contains an air gap in its middle leg. The function of this A.C. excited transformer is to create a magnetic flux in which electrons are accelerated almost to the speed of light in a doughnut shaped accelerator tube which surrounds the electromagnet. The flux produced by the field varies rapidly in shape from the injection stage, when electrons are injected from a cathode filament, through four following stages of deflection and transfer into the magnetic field, a packing phase, a distribution phase, and a phase when the flux is constricted, this leading finally to the emission of elect-



rons which strike a platinum target and thus give rise to extremely high energy X-rays. During this process the electrons travel approximately 1,000 miles.

The magnet responsible for this process, has two rotation-symmetrical poles with parallel faces arranged as a two-legged yoke, the distribution of the field produced being governed by pole pieces which are clamped between the poles. These pole pieces are critically important in that they govern the location of the orbit of the flux and the strength of the magnetic force which stabilises it. Both the pieces and the surrounding accelerator tube are pneumatically pressed into contact with the magnetic yoke, which gives stiffness and protection during transport, in addition to damping down both transformer noise and vibration.

The accelerator tube is constructed of a ceramic known as frequenta, the inner wall of this doughnut tube being coated with a metal film. The materials used are degassed and the tube is evacuated to the low pressure of 10^{-6} torr. The tube is sealed by a 30 micron thick nickel foil, through which electrons accelerated in the flux by the magnet may leave if the production of free electrons is required. For X-ray work, however, these electrons are arranged to graze a platinum target which is fixed to the inner wall of the tube, the resultant X-ray beam being taken out through the frequenta wall of the tube without a window.

The magnet coils are energised by a regulating transformer connected directly to a 50 cycle 3-phase mains supply, the consumption amounting to approximately 12 kVA., and a regulator is employed to correct mains voltage fluctuations between 180 and 240 volts. The betatron is operated from a small portable control case, and with a total weight of under 3½ tons it may be readily transported by truck from site to site.

The substantial savings in exposure time which can be made with this new tool are shown for example by the fact that exposures for steel thickness of between 6 and 8 ins. are only a few minutes, whilst that for a 16 in. thickness of steel is between one and two hours as compared with an exposure of several days which would be required with a supervoltage X-ray tube of the conventional type. Apart from this saving, a new technique of radiographing a workpiece whilst still hot is opened up, and since the cooling-for-examination stage is one where flaws may develop, the elimination of this stage in thick section fabrication will lead to the prevention of costly flaws in this type of work, as further fabrication can follow inspection without the need for a fluctuating temperature gradient. The third point of note is the fact that a magnification technique, giving an image of up to three times the size of the flaw, can be used with this betatron.

Pantak, Ltd., Vale Road, Windsor, Bucks.

Duorider Balance

In recent years the research and development establishment of Griffin & George, Ltd., has been engaged in overcoming the several obstacles to the provision of first-class balances at reasonable cost, and the new Griffin Duorider balance marks another step forward in this new approach to the design of general purpose balances with analytical features.

The new balance has a shortened limb for easier weighing, combined with a single limb bow eliminating impedance; an inclined pointer scale rising with the pointer to facilitate reading and reduce parallax fully relieved agate knives; an easily and rapidly positioned pillar; a rider bar which eliminates fractional weights; and chemically stable, non-magnetic, easily cleaned stainless steel pans. The name of the balance derives from the fact that the metal rider-bar, chemically etched and individually anodised and dyed black, with white-filled graduations, carries two captive riders of 1·0 and 0·1g., respectively, moving over 9 notched divisions from left-hand zero to centre and over 50 divisions from centre zero to the right. The capacity of the balance is 250g. and the sensitivity 2 mg. The finish is plating or hammer-finish for all metal parts except pans and rider bar, and the base is of moulded plastic.

Griffin & George, Ltd., Ealing Road, Alperton, Wembley, Middlesex.

Pure Metaloids

Johnson, Matthey & Co., Ltd., are now producing very pure arsenic, antimony, tellurium and bismuth on a commercial scale. By means of newly developed techniques, arsenic is being produced with an impurity level of the order of 1 p.p.m., detected by use of present known and accepted spectrographic methods. The element is in the form of crystalline lumps and is packed in vacuum-sealed glass tubes. Antimony, with a maximum metallic impurity content of 5 p.p.m., and bismuth, with a maximum level of metallic impurities of 10 p.p.m., are being produced in ingot form. Zone-refined tellurium, with a metallic impurity content of the order of 1 p.p.m.,

is also available in bulk, in the forms of ingot or grain.

Of increasing importance to the semiconductor industry, these four high purity metals find growing application in the form of intermetallic compounds. For example, gallium arsenide is used in the production of special diodes and transistors, indium antimonide and indium arsenide are used for magneto-sensitive devices, and bismuth telluride is used in Peltier cooling units.

Johnson, Matthey & Co., Ltd., 73-83 *Hatton Garden, London, E.C.*1

New Range of Simple Scanners

FILLING the gap between the electronic strip chart recorder and complex data handling systems, a range of simple scanners is announced by Honeywell Controls, Ltd. The new units, of British design and manufacture, will scan process variables at either two or seven points a second. They contain no moving parts (except one gold-plated uniselector), and since slidewires and servo-motors are eliminated, longer life is obtained. There is no " print out "—only an alarm and an annunciator panel using Nixie lights to indicate points scanned and points off-normal. The new scanners are thus ideal for process industries where permanent records are not required and only off-normal points are of interest.

Known as Types B, C and D, these units use the principle of comparing incoming voltage against a preset alarm level, in a non-indicating device called a blind amplifier. An ElectroniK indicator will be included for setting up alarm levels and monitoring single points. Optional facilities include flashing alarms, recycling, alternative input switches and a means of varying the number of input points and set points.

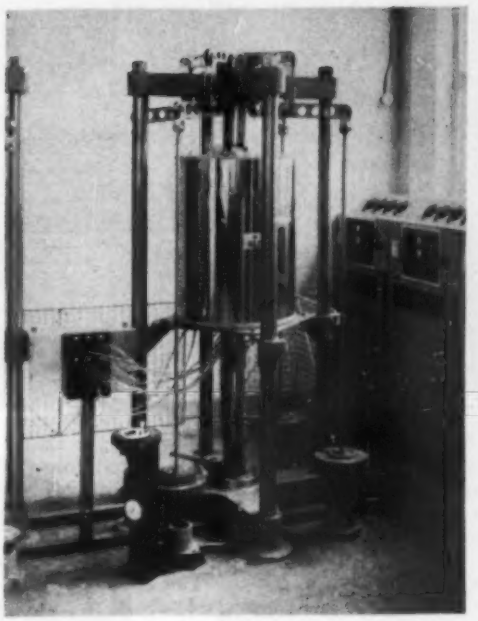
Type B, accurate to $\pm 40\mu$V., scans at two points a second and has a common red light and Klaxon alarm for each group of twelve points. Setting is made by means of ten-turn analogue potentiometers. The scanner stops when an off-normal point is detected, and re-commences on manual restart. Type C is similar in principle but has one alarm light per point and a " memory " to mark the fault while scanning continues. Type D, accurate to $\pm 10\ \mu$V., is much faster at seven points a second, has one red light per point and a " memory." It incorporates a pinboard method of setting alarm levels based on techniques used in full-scale data handling systems.

All the new scanners will be constructed of drawer units to fit a standard relay rack approximately 7 ft. high. The design includes plug-in drawer units and lift up trays for ease of maintenance. The new simple scanners are additional to the existing Honeywell data handling equipment, and to scanners with slidewires and motors.

Honeywell Controls, Ltd., Ruislip Road East, Greenford, Middlesex.

Multi-Specimen Creep Testing Machine

THE research and development department of The United Steel Cos., Ltd., has designed a compact creep testing machine which is now being built and marketed by Distington Engineering Co., Ltd. It is expected to have many applications in industries concerned with the design and manufacture of steel parts for service conditions in steam boilers and turbines, gas turbines and



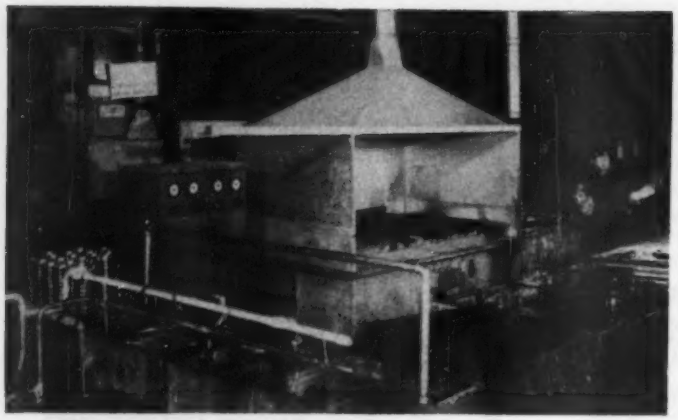
certain chemical plant. Known as the Unisteel multi-specimen creep testing machine, it is designed to accommodate either four high-sensitivity tests or twelve stress-to-fracture tests on the one machine with one furnace and one temperature controller. The machine incorporates four loading systems, each of which caters for one high-sensitivity test or three stress-to-fracture tests in series. Many years of experience in the use of creep testing machines have gone into the design and construction of the new model, a special feature of which is the concentration of a number of testing stations within a comparatively small space. The machine occupies a floor space of only slightly more than one square yard and can be installed in rows at 4 ft. centres. This compares very favourably with the usual single specimen machines, while the machine cost per test point is competitive with other machines on the market. For compact installations, guards can be fitted to prevent accidental contact with the loading weights.

The Unisteel machine can be used for the application of loads up to three tons on a specimen. The complete unit comprises the creep testing machine itself ; a furnace consisting of a fused-silica tube wire-wound in three zones for resistance heating, encased in a polished stainless steel cylinder ; a control panel, incorporating an electronic temperature controller ; and two auxiliaries —a ratchet spanner and lifting jack—which can be used with a number of machines.

Distington Engineering Co., Ltd., Workington, Cumberland.

Cassel Ajax Salt Baths

FIVE new types of electrically heated units are now being added to I.C.I.'s range of oil and gas fired Cassel salt bath furnaces. Made by the company's General Chemicals Division and sold both in this country and in a number of overseas territories under exclusive licence from the Ajax Electric Company, Philadelphia, U.S.A.; they are available in various sizes.



Three of the new types, known as the Cassel Ajax S.R., S.S. and Q., are electrode-heated furnaces with rectangular pots. The salt in the pots is heated by current passing between electrodes immersed in the salt, and the life of the pot is therefore far longer than that of pots heated externally by gas or oil. Continuous operation is recommended. The S.R. furnace is used for heat treatment with neutral salts in the temperature range 700–1,000° C.: the pot is made of interlocking refractory bricks and should last from one to three years. The S.S. furnace is used for carburising with various salts containing cyanide in the temperature range 650–1,000° C. In this case the pot is made of mild steel plate and should have a life approaching one year. The Q furnace is used for quenching and secondary hardening of high speed steel in the temperature range 500–700° C., and the pot of mild steel plate should last up to two years.

The two remaining Cassel Ajax furnaces, the T. and T.I.Q. types, for lower-temperature working, are fitted with immersion heaters. Here the heat is produced in resistance elements immersed in the salt and suitably protected. So, in these furnaces also, the pot does not have to transmit heat, and should last several years. The Ajax T. furnace, used for tempering steel and heat treating aluminium alloys in the temperature range 160–400° C. (special furnaces for temperatures up to 500° C. are available), has a pot made of mild steel which should last four to five years. The T.I.Q. furnace is used for quenching steel during austempering or martempering treatments, and agitation of the salt bath is arranged so that parts being quenched are exposed to a uniform flow of molten salt which extracts heat rapidly and with minimum distortion of the work. The pot is air cooled externally to prevent the salt rising in temperature. If parts have just been austenitised in a high temperature salt bath, a separating chamber is provided in which the high temperature salt is removed.

*Imperial Chemical Industries, Ltd., Imperial Chemical House, Millbank, London, S.W.*1.

High Range Pressuretrol

A RECENT addition to the Honeywell range of pressure controllers is the new industrial Pressuretrol, model P428, designed for general applications requiring control or limiting of gas or liquid pressures up to

3,000 lb./sq. in. It can operate alone as a controller or with other instruments for limit, alarm or signalling service. Five ranges are available, from 30–300 lb./sq. in., to 300–3,000 lb./sq. in.

The industrial Pressuretrol consists of a mercury switch operated by a corrosion resistant Bourdon tube, enclosed in a heavy steel case which resists dust, weather and corrosion. Pressure setting knobs are conveniently located on the front, and tamper-proof knobs can be provided if desired. Switch action is SPST, make or break on pressure rise, depending on the model. The position of the mercury switch is visible through a window in the front of the case. An explosion-proof model is available for use in hazardous atmospheres.

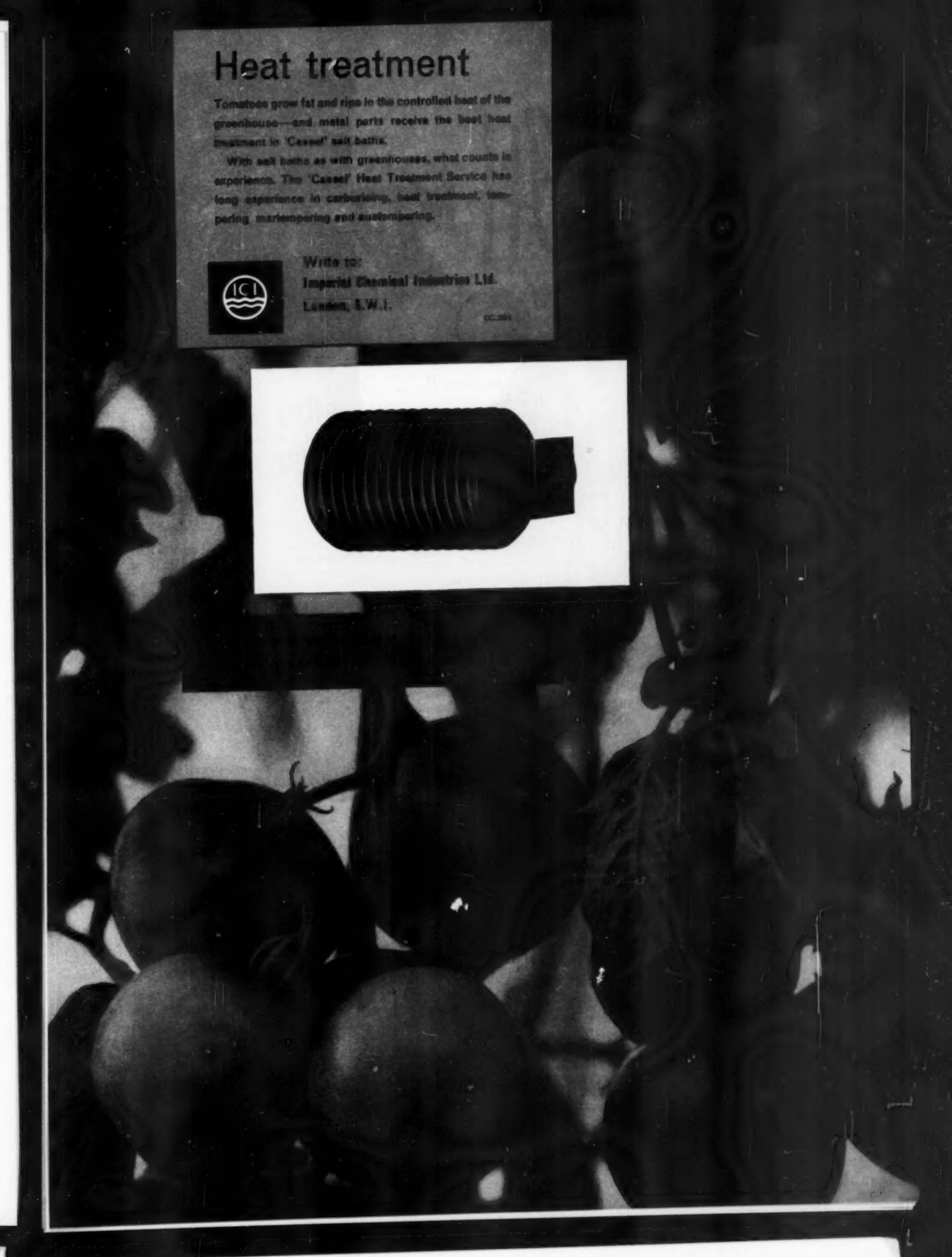
Honeywell Controls, Ltd., Ruislip Road East, Greenford, Middlesex.

Oxygen-Free High-Conductivity Copper Anodes

OXYGEN-FREE high-conductivity copper anodes will shortly be available in this country through W. Canning & Co., Ltd. These anodes have been successfully used in the U.S.A. for several years and have only just become available in the U.K. A number of advantages over extruded or electrolytic copper anodes are claimed. The absence of oxide means that there is more copper for plating available per unit volume of anode than with other types, and that the amount of sludge formed is extremely small. In properly controlled cyanide solutions the anode surface is completely free of film and reveals the characteristic large crystal plate formation. Furthermore, the uniform characteristic crystal structure permits the anodes to dissolve uniformly with complete absence of under-cutting or graining. The uniform dissolution at solution level as well as below the surface enables the anodes to be used until they have almost completely dissolved. The combination of these factors makes for improved deposit quality.

Oxygen-free high-conductivity copper anodes will be supplied in an oval cross section of 1½ in. by 3 in. and in standard lengths up to 54 in. long.

W. Canning & Co., Ltd., Great Hampton Street, Birmingham, 18.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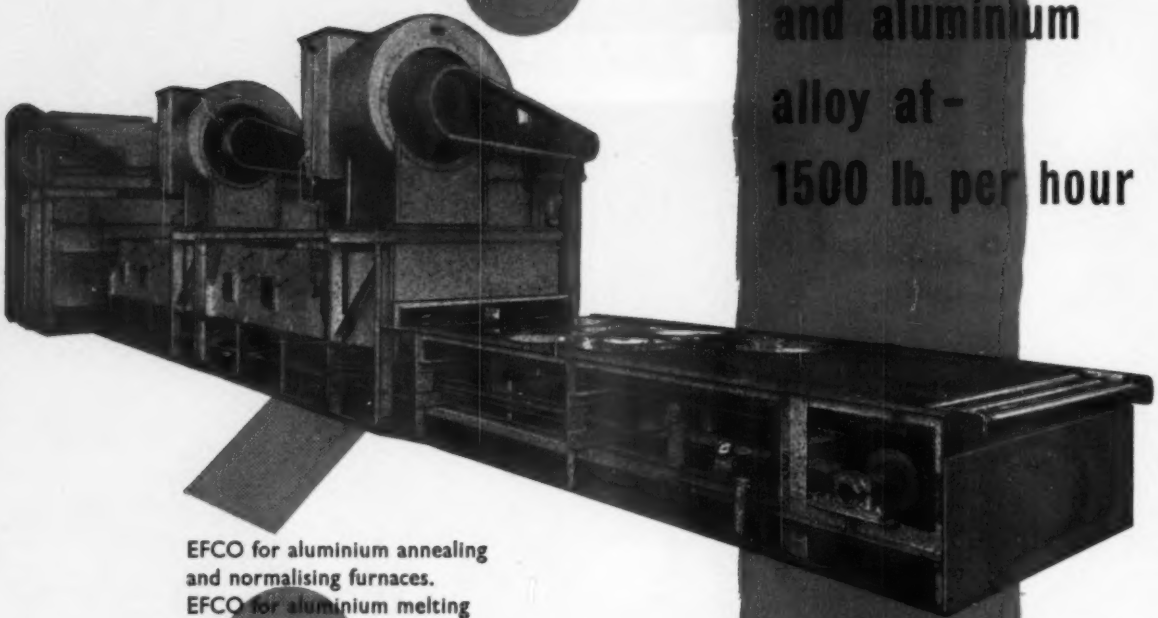

Efco-Lindberg flash annealing furnace installed in the Rolling Division of High Duty Alloys Ltd. for [illegible] of sheets and circles. The furnace, rated at 600 kW, has a heating chamber 20ft. long and a cooling table 30ft. long.

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LABORATORY METHODS
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INSTRUMENTS AND MATERIALS
NOVEMBER, 1960 Vol. LXII, No. 373

A Rapid Accurate Method for the Determination of Silicon in Carbon and Low-Alloy Steels and Cast Irons

By C. G. Westwood, L.I.M.

It is claimed for the method that it is more rapid than any of the extant accurate methods and more accurate than any of the extant rapid methods. The reliability has been proved by many hundred estimations during the development of the method, and it has now been in use with complete success for some months, in the laboratories of two commercial iron foundries. It has been applied to cast iron and carbon and low alloy steels ; it has not yet, to the author's knowledge, been applied to higher alloy steels and cast irons, but subject to normal interfering elements, there seems no reason why the method should not be extended to these materials.

THE method to be discussed here is in no way new in fundamental concept, but originated from a consideration of the well-established modified Regelsberger method[1-6] employed for the determination of silicon in aluminium-silicon alloys. It is well-known that in this modified procedure the conventional double evaporations necessary in the classical Regelsberger method[7] are avoided. This is accomplished by the addition of a specified amount of gelatine at a critical stage in the method, resulting in the conversion of orthosilicic acid from a hydrosol to a hydrogel of silicic acid, in a single step and without any true dehydration being carried out. This process is termed gelatine flocculation, and by its use lengthy double evaporations are avoided ; the method is very much simplified, and a considerable time saving results. In addition to this, further advantages are claimed for the use of the modified Regelsberger method, and these will be discussed in some detail at a later stage.

The gelatine flocculation technique does not seem to have been applied to the determination of silicon in ferrous materials to any great extent. To the author's knowledge there has been no recent published work on this topic. However, gelatine has been used[8] as a coagulating agent to facilitate the filtration of silicic acid in ferrous and non-ferrous materials after dehydration of the orthosilicic acid has been achieved by conventional fuming with sulphuric or perchloric acids. There are, of course, several methods readily available for the gravimetric determination of silicon in ferrous materials. These include both single and double evaporation methods, but in each, complete dehydration of the orthosilicic acid must occur in order to obtain quantitative results. The single evaporation methods, which may be of either the " single baking " or the " single fuming " type, make up the bulk of the quicker methods in general laboratory use. The main disadvantage of these methods is the comparatively low results obtained as compared with the more lengthy double evaporation methods. This point is well illustrated by reference to the silicon figures quoted in Table I, for the hematite iron (B.C.S. No. 236) used in this investigation : it will be noted that the differences quoted between double and single evaporation methods are appreciable. Silicon figures obtained by numerous analysts using single evaporation nitro-sulphuric acid methods are, on the average, 0·08% lower than figures obtained by the double evaporation perchloric method (B.S. 1121 : Part 10 : 1948). The merit of the more lengthy double evaporation method where accuracy is of utmost importance needs no further emphasis. It was also noted during the course of the experimental work that, as the percentage of silicon in a given sample increased, so the amount of silicon lost by using a single evaporation method increased proportionally. A high degree of accuracy is often not essential for the bulk of routine work, and single evaporation silicon methods are quite

TABLE I.—RESULTS ON RIDSDALE STANDARD HEMATITE IRON No. 236

Complete Analysis (%)	C	Mn	Si	S	P	Ni	Cr	Mo	V	Ti
	3·07	1·31	2·04	0·051	0·051	<0·02	0·016	<0·01	0·018	0·072

Silicon Percentages Obtained by Standardising Analysts				Author's Results
Analyst No.	Nitro-Sulphuric Acid Method		Perchloric Acid Method	Gelatine Method
	Single Evaporation	Double Evaporation	Double Evaporation	
1	1·95	2·06	2·08	2·06
2	—	2·07	—	2·02*
3	1·98	—	2·02	2·06
4	—	—	2·02	2·06*
5	1·98	—	2·07	2·06
6	—	2·00	1·99	2·08*
7	—	—	2·02	2·00
8	1·92	—	2·00	2·06*
9	—	—	2·01	2·08 2·08*
Average	1·96	2·04	2·03	Average 2·056 or 2·06
All results after HF treatment				No HF treatment except where indicated by *

adequate. The only disadvantage when using double evaporation silicon methods is the time consumed for a single determination, as much as 50% more as compared with a single evaporation method.

It was with the foregoing facts and problems in mind, that the improved gelatine flocculation technique discussed here was developed, to meet the need for a quick and accurate method of determining silicon in pig iron. In the course of an investigation of the desulphurisation of pig iron[9] using solid lime, it was necessary to be able to establish with precision the silicon content of a large number of pig iron samples. At the conclusion of the experimental work some seven hundred pig iron samples had accumulated, and a *precise* silicon figure was required on each sample. This was no mean task, as it was realised from the start that the rapid single evaporation silicon methods available would not be sufficiently accurate. On the other hand, the more accurate double evaporation silicon methods available were far too time-consuming for the large number of determinations involved. It was decided that the method employed for the silicon determinations must give accuracies to within ± 0·02% silicon in order that warrantable conclusions could be drawn. (The only conventional method that could achieve this was the double evaporation perchloric acid method—B.S. 1121 : Part 10 : 1948—which as already stated was unsuitable).

The need for the unusually high degree of accuracy for the desulphurisation problem was that it was desired to examine the part that silicon played during the transfer of sulphur from metal to lime. The amount of silicon associated with this transfer is only small, and it was calculated from the equation that 0·01% of silicon is associated with a transfer of 0·023% sulphur :—

$$2CaO + 2FeS + Si = 2CaS + 2Fe + SiO_2$$

Thus, it was essential that the method of analysis employed should be capable of detecting small differences in silicon content, and should be capable of giving accuracies to within ± 0·02% silicon, i.e. ± 0·046% sulphur.

For these reasons the gelatine flocculation method as applied to ferrous materials, and in particular pig irons with silicon contents ranging from 1·50 to 2·50%, was pursued. The method and its development are described below.

The Gelatine Flocculation Technique

Reference to the literature dealing with the determination of silicon by this procedure shows that in the main, the method is restricted to aluminium alloys, and that it is a further development of the classical Regelsberger method, first introduced in 1891. The use of gelatine as a flocculating agent for the silicic acid was developed in Germany during the second world war, to save time when determining silicon in the vast amount of light alloys used in the aircraft industries : it proved to be wholly successful. The modified Regelsberger method became popular in the United Kingdom and can be found in most textbooks dealing with the analysis of aluminium alloys.

The salient features of the modified Regelsberger method are as follows. First, the simplicity and considerable time saving achieved by its use, coupled with the very important fact that the flocculation of the silicic acid is quantitative. Second, if washing of the flocculated silicic acid is carried out carefully and correctly no subsequent hydrofluoric acid purification of the silica precipitate obtained after ignition is necessary. The modified method was found to cope easily with the high silicon aluminium alloys, and results obtained on these were found to be consistent and could be interpreted with confidence. The classical Regelsberger method may be compared with the standard methods in use for the determination of silicon in ferrous materials, in which dehydration of the orthosilicic acid is accomplished by fuming in sulphuric or perchloric acid. However, as previously stated, this process does not give quantitative results on a single dehydration, and if quantitative results are to be obtained a double dehydration technique must be employed. This makes these methods lengthy and tedious. In the modified Regelsberger method the conversion of orthosilicic acid from a hydrosol to a hydrogel is made complete by agitating the final solution vigorously, after the addition of a specified amount of gelatine solution. Thus, quantitative silicon results are obtained, without fuming or baking, and in a single step. This process is termed flocculation and produces a hydrogel of silicic acid which is readily filterable. The exact mechanism and chemistry of the flocculation process is not clearly understood. Analysts who have used this modified method, however, will agree that the method requires more attention to detail than conventional silicon methods if accurate reproducible results are to be obtained. This attention, is however, more than justified by the advantages obtained in the conversion of a long tedious method to a relatively short and simplified procedure. It can be appreciated, therefore, that with the successful application of this same flocculation technique to the silicon determination of ferrous materials, a rapid, accurate and simple method might result.

Experimental Work

The experimental work was initiated by conducting relatively simple trial tests on a hematite iron (British Chemical Standard No. 236), the full chemical composition of which is given in Table I. This iron was chosen for the trials because :—

(*a*) it had a silicon content and chemical composition of the order desired ; and

(*b*) the certificate of analysis issued with the iron gives comparative figures for silicon which have been obtained by three standard methods, one of which is a British Standard method (i.e. B.S. 1121 : Part 10 : 1948—Double Evaporation Perchloric Acid Method) : this method is claimed to give consistent accuracies to within ± 0·02% on silicon contents up to 4·00%.

Samples of hematite iron ranging from 1g. to 4·673g. were dissolved in hydrochloric-sulphuric acid mixture of a similar strength to that employed in the final stage during the determination of silicon in aluminium alloys using the modified gelatine method. When the samples were dissolved the solution was oxidised with dropwise additions of nitric acid, boiled down to a low bulk, cooled slightly and a solution containing 0·1 g. of gelatine added. This solution was agitated with a rubber-tipped "bobby," allowed to stand, and finally filtered and washed in the conventional manner. All results obtained were discouragingly low and inconsistent. Furthermore, silica precipitates obtained

after ignition were all contaminated, mainly with iron. The procedure was repeated with fresh samples, but this time the final solution was boiled after the gelatine addition for five minutes, cooled, agitated and then filtered and washed as before. Once again the silicon results obtained were low and all silica precipitates were contaminated. It was discovered later that raising the temperature of the final solution above 80° C. after the gelatine addition renders the gelatine partly ineffective and quantitative results cannot be obtained.

The initial attack employed was found to be unsuitable for two reasons :—

(1) the rate of attack using the hydrochloric-sulphuric acid mixture was too slow ; and

(2) it seemed obvious from the results obtained that the method of approach was not satisfactory.

A new method of attack was therefore considered. The reluctance in the early stages of the work to use acid mixtures containing nitric acid will be noted. This was mainly due to an attempt to reproduce the exact conditions of the final acid solution used in the modified Regelsberger method. Obviously, if an acid mixture containing nitric acid had been used, the final solution would have needed fuming fairly strongly to remove the nitric acid. In doing this the object of the flocculation technique would have been defeated, i.e. dehydration of the orthosilicic acid would already have been partly accomplished. It is to be noted that the modified Regelsberger method does not make use of nitric acid : oxidation is achieved by the use of hydrogen peroxide. It was decided to use peroxide in future trials instead of nitric acid. At this point it could be seen that by trying to adhere rigidly to the exact conditions arising in the modified Regelsberger method little headway was being made, due mainly to the strong dissimilarities of the two alloys. Even the method of initial attack is dissimilar, aluminium alloys being attacked with a strong alkali and ferrous materials with mineral acids : thus, the nature of the salts present in the final solutions prior to dehydration bear no resemblance at all. It was therefore decided to find the acid or acid mixture that would give the highest rate of initial attack for the pig irons and commence from there, disregarding completely conditions arising in the modified Regelsberger method.

After trying a variety of acids and acid mixtures for the initial attack, it was discovered that nitro-sulphuric acid mixture of critical strength gave the highest rate of initial attack using a standard sample weight and iron. The method first tried was then repeated again ; this time, however, the sulphuric-hydrochloric acid mixture was substituted by nitro-sulphuric acid. Silicon results obtained were much improved, but still low, and ignited residues obtained were still slightly contaminated and required purification by standard hydrofluoric acid treatment. Contamination of this type suggested bad washing practice, but this was not considered to be the case. Care was exercised during washing operations and the 50% hydrochloric acid and water used were very near to boiling point. If anything, the precipitates were being overwashed. At this stage of the investigation a new washing technique was suggested to the author.[10] This consisted in washing the precipitated gelatinous silicic acid with hot 5% oxalic acid. This technique ultimately turned out to be so efficient that the old hydrochloric acid-water washing procedure was completely abandoned. It was observed that by using the oxalic acid wash no further contamination of silica precipitates due to iron was experienced. From this it was concluded that in ferrous materials the high concentration of ferric chloride present after taking up dehydrated residues with hydrochloric acid is an important factor. Also, the same difficulty is to be expected if hydrochloric acid is used as a wash for the removal of iron from a solution containing flocculated silicic acid but no hydrochloric acid (i.e. in a method where no hydrochloric acid is used at all because there is no fumed residue to take up ; dehydration is substituted by flocculation). It must be made clear that the precipitated silicic acid brought about by gelatine flocculation is more gelatinous and prone to coagulation than those obtained by conventional dehydration methods. The precipitate is therefore more susceptible to contamination unless the necessary washing precautions are observed. For the above reasons the use of hydrochloric acid as a washing agent for this proposed new method was abandoned. Moreover, other reasons for condemning its use were found, and the ultimate method developed from this investigation does not require the use of hydrochloric acid in *any* stage.

It should be mentioned here that the reason why no contamination due to iron was experienced in the modified Regelsberger method in which bulky gelatinous precipitates are handled is obviously due to the fact that the percentage of iron in aluminium alloys is small and rarely exceeds 1·00%. It is well-known that freshly precipitated silicic acid has a measurable solubility in certain mineral acids which cannot be totally eliminated, but can be reduced if certain precautions are observed. Hydrochloric acid has a fairly high solubility for freshly precipitated silicic acid, particularly if the acid is concentrated. Hydrochloric acid has a wide application for taking up fumed or baked residues in silicon and silica determinations prior to the filtering operations. If, however, the hydrochloric acid used is dilute the error that can be introduced by this means is small. It was stated by Hawley[11] that by far the largest and most troublesome error that arises in silicon determinations is due to the solubility of freshly precipitated silicic acid in the hydrochloric acid. This error is much greater than most chemists realise. The amount of freshly precipitated silicic acid that dissolves in hydrochloric acid depends on the following conditions. First, the amount of acid present ; second, the strength of the acid ; third, the temperature ; and fourth, the length of time the silicic acid is in contact with the acid. These are the principal conditions governing the solubility of silicic acid in hydrochloric acid, though there may be others. By far the most important is the amount of acid used. Most chemists use acid of about the same strength to treat the dehydrated silicic acid, and the determinations are brought to a boil, ensuring the same temperature. Few, however, consider the importance of using a definite amount of acid. It seems to be a prevalent idea that the amount of silicic acid dissolved by the acid solution is proportional to the amount of silicon present in the sample. This is certainly not the case. Experiments prove clearly that when only small amounts of silicic acid are present, the amount dissolved is proportional to the quantity of acid solution present and not to the amount of silicon in the sample. It may seem to the reader that

this point has been overstressed, but when an accurate method is being developed, traces of silica which are lost at progressive stages in a method become important. For example, it is often found that the amount of silica recovered during a second evaporation in standard methods does not normally exceed a few milligrams if a sound laboratory technique is employed. This amount of silica can easily be lost by solution of the freshly precipitated silicic acid after dehydration in an unsuitable mineral acid or strong solution of a mineral acid used to take up the dehydrated residue. For this reason the strength of hydrochloric acid to be used for taking up dehydrated residues in the British Standard method for silicon determinations is specified and should be kept constant. The following figures illustrate the relevant approximate solubilities of freshly precipitated silicic acid in various commonly used mineral acids.

	Silica–mg.
Nitric Acid	1·5–2·0
Aqua Regia	1·0–1·5
Hydrochloric Acid	0·5–2·0
Sulphuric Acid	0·5–0·75
Perchloric Acid	0·1–0·5

It can be seen that the two most favourable acids to use are sulphuric and perchloric, but the above figures are not maximum solubilities. These may be exceeded if an unsatisfactory technique is employed. It was for these reasons, and other points brought out in the previous discussion, that the method to be described uses only nitro-sulphuric acid, with a boost oxidation in the early stages with hydrogen peroxide. It will be noted later that the silicic acid is flocculated in a solution that is mainly sulphuric acid.

It has already been indicated that the first signs of success with this new approach were obtained when the samples of pig iron were attacked in nitro-sulphuric acid of a specified critical strength. The rough outline of this method is as follows. The samples were first attacked in nitro-sulphuric acid, and after complete solution a quantity of hydrogen peroxide was added. The peroxide addition was found to be an important factor in the successful accomplishment of this method. It was found that rapid and complete oxidation of the silicides in the iron at an early stage was very important, and the peroxide adequately filled the role of the oxidant. Directly the peroxide had done its work the solution was cooled and the gelatine added. The solution was then agitated as before, filtered, and washed with the oxalic acid. Silicon results obtained by this procedure were now more encouraging and were comparable with those obtained by single evaporation methods (for example, silicon figures obtained on British Chemical Standard hematite iron No. 236 averaged at 1·92%). The figures obtained were calculated from direct weights of silica, without hydrofluoric acid purification. This was because the silica precipitates after ignition were extremely white and looked to be free from contamination. Random samples were given the hydrofluoric acid purification treatment and the weights of contamination did not exceed 0·0002 g. of silica, which is equiv. to 0·0094% of silicon on a 1·00 g. sample. In the ority of cases the weight of impurity was too small t detected.

The cr. point in the development of this new method came when it was discovered that to gain the full benefit from the gelatine flocculation process it was essential that incipient precipitation of the silicic acid must have commenced naturally in the acid solution, and be at a fairly advanced stage before the gelatine is added. It was at this stage in the work that the importance of temperature control during the gelatine flocculation process was found. If the temperature of the final solution exceeded 70–80° C., low results were experienced; from this it was considered that at temperatures above this the gelatine becomes partially ineffective. This statement was not confirmed, but as long as temperature control was exercised no difficulties or low results arose. It was conclusive, therefore, that temperature control was essential if quantitative reproducible results were to be obtained. The outlines of the final method which proved wholly successful are now given.

The sample of iron is attacked in the nitro-sulphuric acid mixture and towards the final stages of dissolution the peroxide addition is made. Following the peroxide addition the solution is boiled down fairly rapidly until incipient precipitation of the silicic acid is visible. The solution is held at this stage until the precipitation of the silicic acid is at a fairly advanced level. This stage can be judged very accurately. As soon as the solution commences to evolve clearly visible fumes of SO_3 the correct stage for removal has been reached. This step is the most crucial one in the whole process, and the success of the method depends upon it. If the solution is not removed at the precise moment and evaporation of the solution is carried too far, there is a danger of sulphates being precipitated from the concentrated solution, which means that the estimation has been ruined. Obviously, as no acid is used after evaporation in this method, any sulphates formed due to over evaporation of the solution will not be completely taken back when the gelatine solution is added. In fact there is little chance of any sulphates that are formed going back into solution at all, because during the flocculation process the solution cannot exceed 80° C. Also, any sulphates present in the solution prior to the filtering operation will not be removed during the filtering and washing process.

After the critical stage just described has been passed successfully, the evaporated solution is cooled slightly and the gelatine solution added. After the gelatine addition has been made the resultant temperature in the solution should be between 60 and 70° C. The solution is then agitated vigorously with a rubber-tipped " bobby " to form a froth and to stir in as much air as possible, which favours the flocculation of the silicic acid. A minimum specified standing time of ten minutes is important, and should be strictly observed if extremely accurate results are to be obtained. However, if the solution after " bobbying " is filtered immediately, a result of fairly high accuracy can be obtained which in nine out of ten times is very near to the accuracy obtained by double evaporation conventional methods.

It was observed in a series of test determinations that the gelatine flocculation process resulted in a very pronounced coagulation of the silicic acid, which facilitated filtration and washing. The ignited silica residues obtained were consistently white and of high purity. Random samples were again given the hydrofluoric acid treatment but the contamination experienced was not of sufficient magnitude to warrant any adjustment to silicon percentages obtained. Results obtained

TABLE II.—RESULTS ON BRITISH CHEMICAL STANDARD HEMATITE IRON No. 236/1

Complete Analysis (%)	C	Si	Mn	S	P	Ni	Cr	Mo	V	Ti
	2·83	1·78	1·13	0·050	0·066	<0·02	0·02	<0·01	0·02	0·065

Silicon Percentages Obtained by Standardising Analysts		Author's Results by Gelatine Method
Analyst No.	Result	
1	1·77	1·78
2	1·77	1·72
3	1·78	1·76
4	1·78	1·78
5	1·78	1·78
6	1·76	1·80
7	1·78	1·76
8	1·78	
Average	1·78	Average 1·768 or 1·77
Analyst No. 2 used the nitro-sulphuric acid method with double evaporation. The remaining analysts used the British Standard perchloric acid method (B.S. 1121 : Part 10 : 1948). All ignited the precipitate and treated it with HF.		No HF treatment

TABLE III.—RESULTS ON BRITISH CHEMICAL STANDARD STEEL No. 239/1

Complete Analysis (%)	C	Si	S	P	Mn	Ni	Cr	Mo	V	Cu	As	Sn
	0·32	0·27	0·025	0·024	0·69	0·193	0·18	0·03	< 0·005	0·48	0·033	0·06

Silicon Percentages Obtained by Standardising Analysts		Author's Results by Gelatine Method
Analyst No.	Result	
1	0·265	0·280
2	0·274	0·282
3	0·260	0·276
4	0·281	0·269
5	0·285	0·282
6	0·265	0·280
7	0·266	
8	0·280	
Average	0·270	Average 0·278 or 0·28
Analyst No. 8 used the standard photometric method B.S. 1121 : Part 15 : 1949. The remaining analysts used the standard gravimetric method B.S. 1121 : Part 10 : 1948. Analysts 3 and 7 also used the standard photometric method and found 0·260% and 0·265%, respectively.		No HF treatment

were of high accuracy and were now comparable with silicon results obtained by the British Standard method for silicon determination in ferrous materials (B.S. 1121 : Part 10 : 1948). In fact most of the results obtained on the standard iron gave silicon figures slightly higher than those obtained by the British Standard method. A blank test was conducted with each set of silicon determinations initially, but this was later found unnecessary because no contamination or impurity pick-up was being experienced. It is recommended, however, that a blank determination be made on starting a new supply of gelatine and paper pulp clippings.

Results

The results obtained by this new method on the experimental standard iron are shown in Table I. These were so successful that the same method was tried out on more standard cast irons and steels, the results obtained being set out in Tables I–IV.

TABLE IV.—RESULTS ON BRITISH CHEMICAL STANDARD No. 239

Complete Analysis (%)	C	Si	S	P	Mn	Ni	Cr	Mo	Cu	V	As	Sn	Co
	0·317	0·24	0·047	0·022	0·73	0·23	0·10	0·02	0·31	—	—	—	—

Silicon Percentages Obtained by Standardising Analysts		Author's Results by Gelatine Method
Analyst No.	Result	
1	0·239	0·246
2	0·246	0·245
3	0·235	0·248
4	0·245	0·244
5	0·241	0·241
6	0·235	Average 0·245
7	0·240	
8	0·240	No HF treatment
b	0·239	
Average	0·240	

The Method as Finally Developed

Notes

(1) This method has not been tried on irons and steels of the medium to high alloy type, and interference experienced from elements in conventional methods with a few exceptions is to be expected when using this procedure. Contamination due to lead found in some free machining steels can be effectively removed by the standard ammonium acetate wash, without complication. However, the low alloy irons and steels can be effectively covered by this procedure, and any contamination experienced from the medium alloy steels or irons by particularly troublesome elements can in most cases be taken care of by the standard HF treatment if so desired. It must be emphasised that the procedure to be described here was not developed as a universal silicon method but as a method specifically for silicon determination in pig iron and, later, steels. Its wider application to steels, cast irons of the very high silicon type (acid resisting), steelworks slags, and refractory materials, etc., is well worth consideration.

(2) As already noted, it was essential to keep time per estimation to a minimum. To this end the sample weight for pig iron was standardised at 1 g. and the sample ground extremely fine. This gave a very high rate of initial attack and solution. A 2 g. sample was standardised for steel.

(3) As previously mentioned the condition of the gelatine solution used is very important and critical. The gelatine solution must be of high purity and sterile. It is recommended that a fresh solution be made up every day, and all solutions older than this be disregarded.

Reagents Required

Silicon Acid Mixture—150 ml. of sulphuric acid (S.G.1.84) + 150 ml. of nitric acid (S.G. 1.42) made up to 1 litre.

Hydrogen Peroxide (20 volume strength).

Gelatine Solution (1·00% solution).—Dissolve the gelatine in warm—not boiling—distilled water, make up to volume and use fresh. The gelatine solution can be kept sterile by making small additions of thymol. The thymol in sufficient quantity will prevent microbial growth. If for any reason the gelatine solution should become contaminated or cease to be sterile it will not function correctly.

Oxalic Acid (5% solution in distilled water).

Procedure

Attack the sample in 20–30 ml. of acid mixture in a squat 400 cc. beaker. Simmer gently on the edge of the hot plate until the sample is almost dissolved. Add 10–15 ml. of hydrogen peroxide, and when the effervescence has subsided move the beaker into the hottest portion of the hot plate. Do not force the initial dissolution of the sample by vigorous boiling, or the solution may reach the critical stage too early and before the sample has been fully digested.

Evaporate the solution until the critical stage is

reached, i.e. fumes of sulphur trioxide are just commencing to be evolved. At this stage incipient precipitation of the silicic acid will generally be visible. Remove the beaker from the hot plate, cool slightly, add 20 ml. of warm water, followed by 20 ml. of the gelatine solution. Immediately stir and agitate the solution vigorously with a rubber-tipped "bobby" for not less than two minutes, to form a froth and stir in air. Allow the solution to stand now for at least ten minutes in a warm place. Filter through papers if preferred, but for speed through a medium, ashless paper pulp pad using suction. Remove traces of silicic acid adhering to the sides of the beaker by "bobbying" and washing into the filter with hot distilled water containing 50 ml. of the gelatine solution per litre. Wash the precipitate well (six to eight times) with hot 5% oxalic acid solution and carefully transfer to a fused silica or platinum crucible.*

Ignite the filtered precipitate slowly at first to avoid any losses by mechanical scattering of the silica by escaping gases. Then raise the temperature to 1000° C. or more to expel the last traces of combined water: this is especially necessary with large bulky silica precipitates. After ignition cool the precipitate in a vacuum dessicator, and weigh as rapidly as possible, since approximately 0·5–1·0 mg. of moisture is absorbed during the first minute or so of exposure to the atmosphere. This rapid rate of absorption of moisture decreases considerably after this time.

* A final wash after the oxalic acid/water washing, of industrial alcohol can be used if so desired. It is claimed that by this process the burning off time is reduced.

Acknowledgments

The author's thanks are due to Mr. J. C. W. Davies of Wolverhampton and Staffordshire College of Technology, who first suggested the application of the flocculation technique to ferrous materials: to Mr. G. T. Hume of Wednesbury College of Technology, whose work on the lime desulphurisation of pig iron necessitated the accurate determination of silicon on large numbers of samples and who saw the possibilities of the method applied to his requirements; and to Mr. E. Wood of the Midland Research Co. Ltd., for general advice and encouragement.

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Griffin and George Expansion

IN the short space of three weeks, Mr. K. G. Sinclair, chairman of the Griffin and George group of companies, officially opened new premises at two provincial centres, Glasgow and Manchester. The new Scottish headquarters at Braeview Place, Nerston, East Kilbride, is another step in the programmed expansion and development of the firm founded 134 years ago ten miles away from Braeview Place, in Buchanan Street, Glasgow, by John Joseph Griffin. The firm began as an offshoot of the publishing house, Richard Griffin and Co., and as the result of a book written by John Joseph Griffin describing experiments seen by him at Anderson's University, Glasgow, which ran through six editions and created such a demand for the instruments described in the experiments that Griffin's Chemical Museum was founded to meet it.

The transfiguration of Griffin's Chemical Museum of 1826 into the immense Griffin and George group of to-day is the result of over a century-and-a-quarter of planned expansions which have ever maintained the progressive outlook of the original founder. The business was moved to London in 1848, and in 1926, a hundred years after its foundation, it was merged with a business founded by John Tatlock in Glasgow in 1881, to form Griffin and Tatlock, Ltd. In 1944, the laboratory supply firm founded by Stanley Belcher and Edward Mason in Birmingham in 1906 was incorporated in Griffin and Tatlock, and in 1954, the greatly expanded requirements of modern industry and education led to the merger between Griffin and Tatlock, Ltd., and W. and J. George and Becker, Ltd., to form Griffin and George, Ltd., still based on the principle of scientific service by scientists for scientists as laid down by John Joseph Griffin in 1826. In 1958 the company was reorganised on a group structure, and in 1960 one of the best known manufacturers of microscopes and optical instruments, R. and J. Beck, Ltd., joined the group.

The new Scottish premises will unite the two former centres at Johnston Terrace, Edinburgh and Renfrew Street, Glasgow, which had become inadequate to handle the ever increasing demand in Scotland for the products of the group. Customers in the Edinburgh region will, on calling Caledonian 4868, be connected by direct telephone to East Kilbride at only the cost of a local call.

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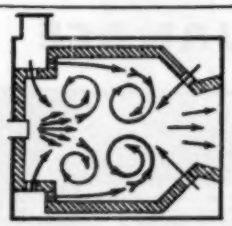

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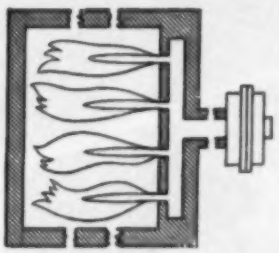

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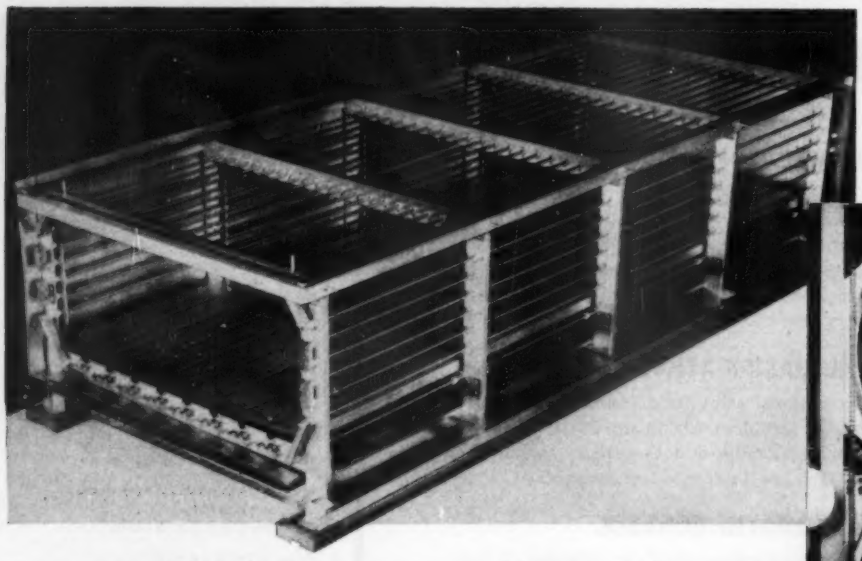

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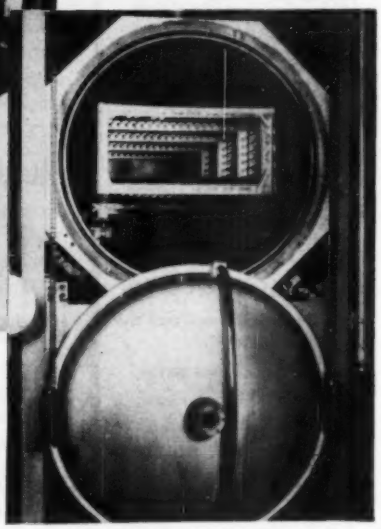

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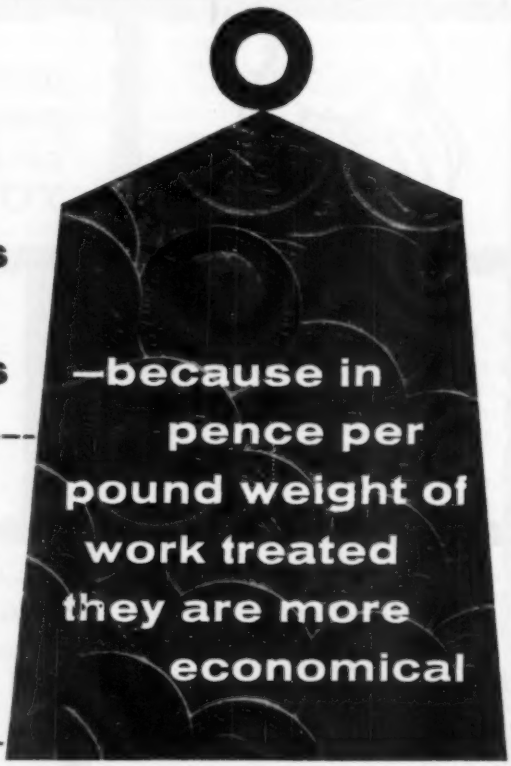

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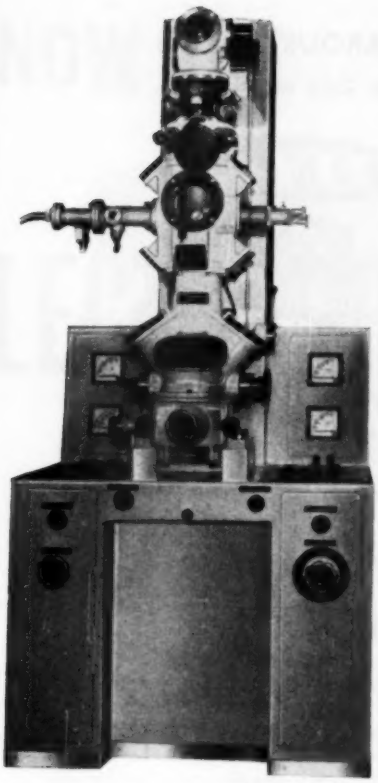

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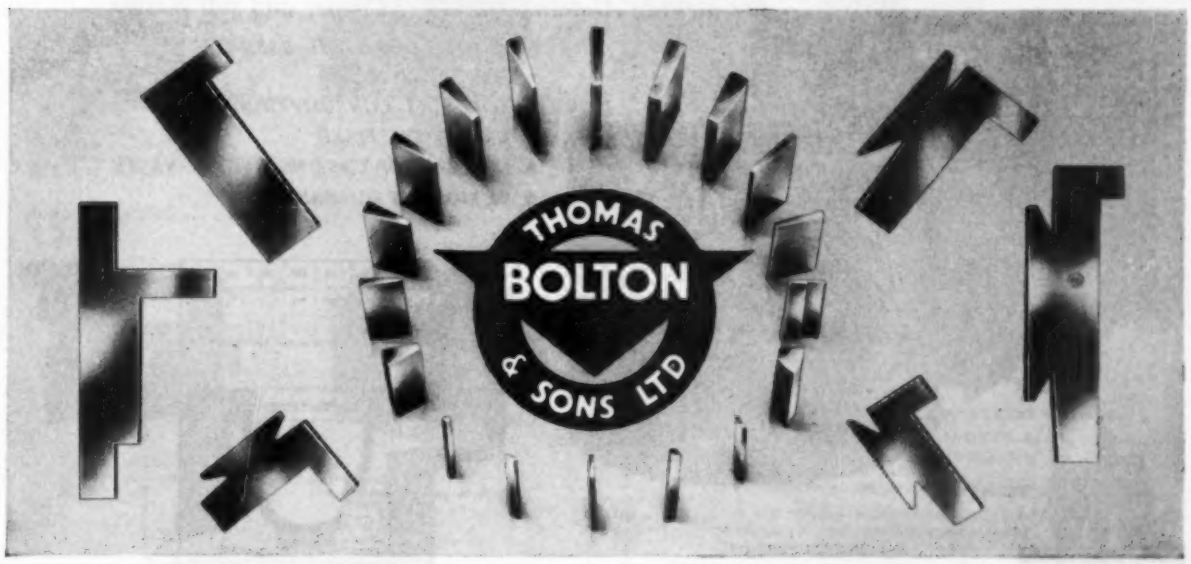

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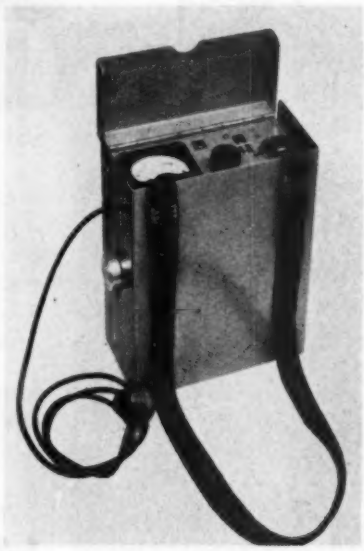

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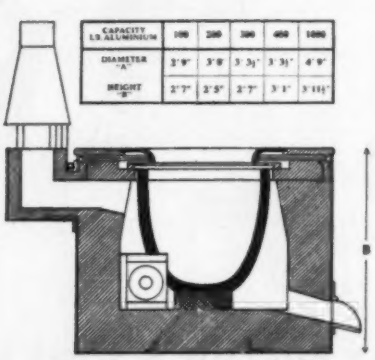

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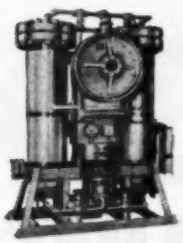

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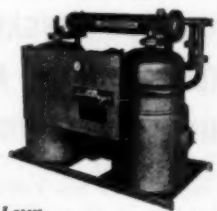

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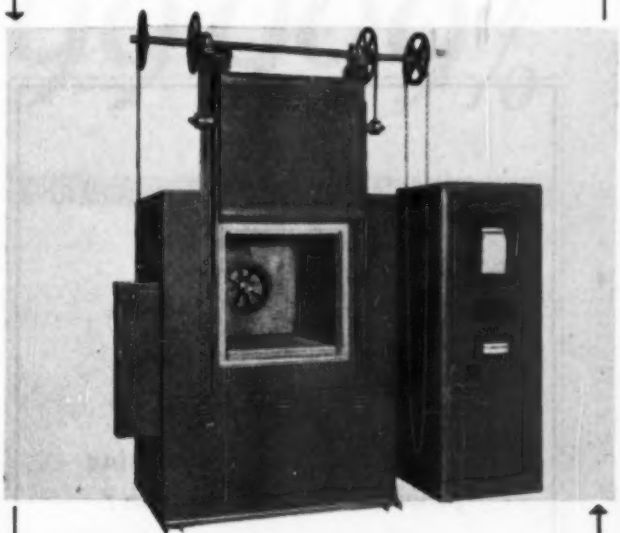

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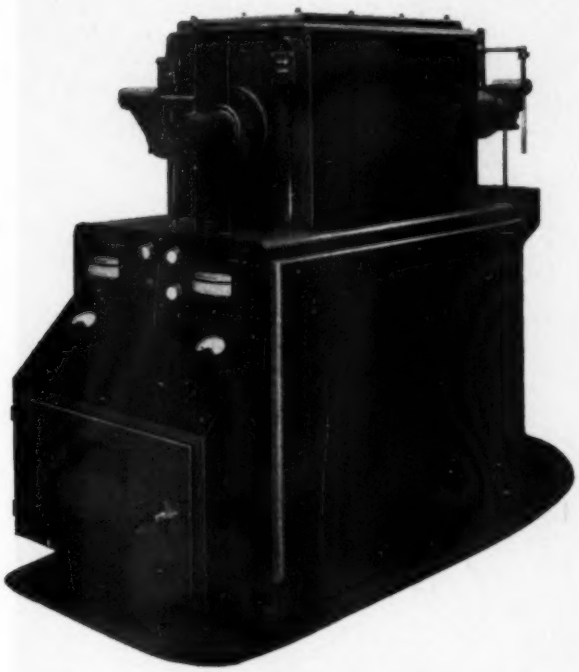

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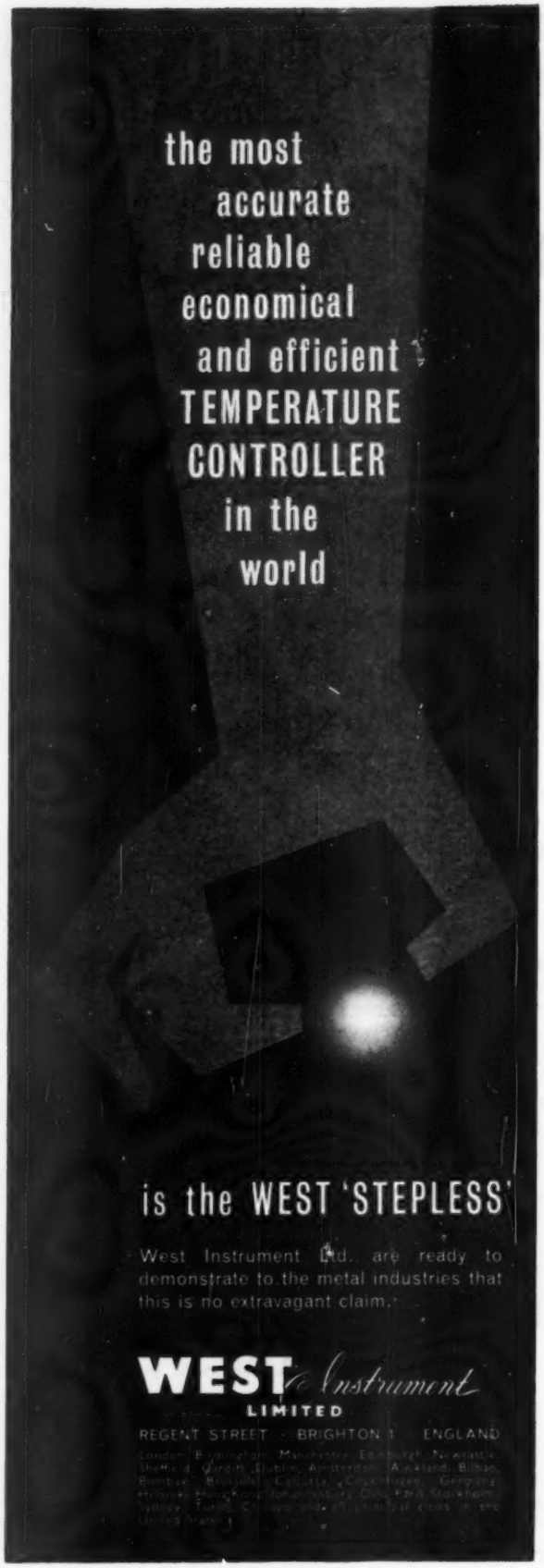

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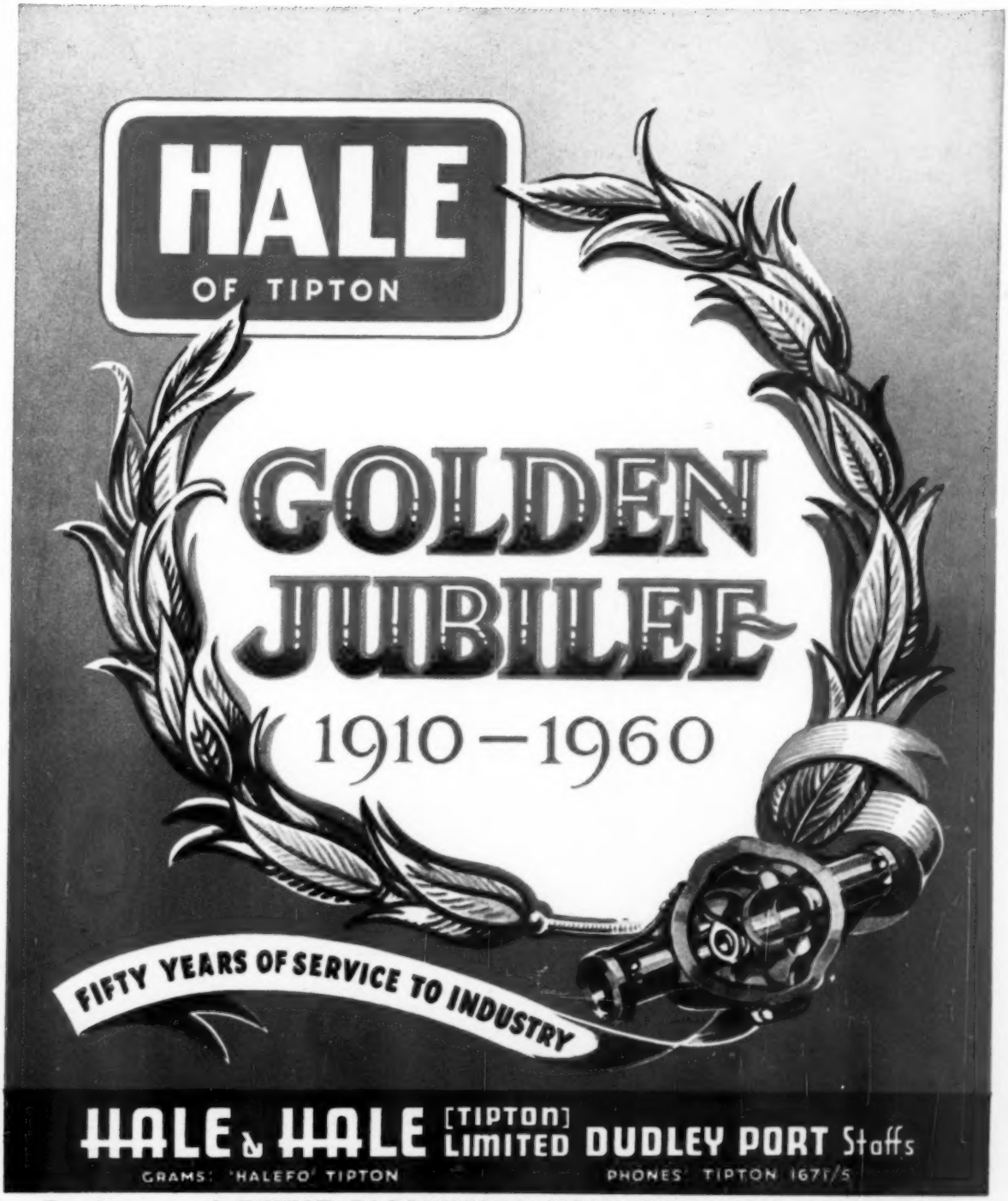

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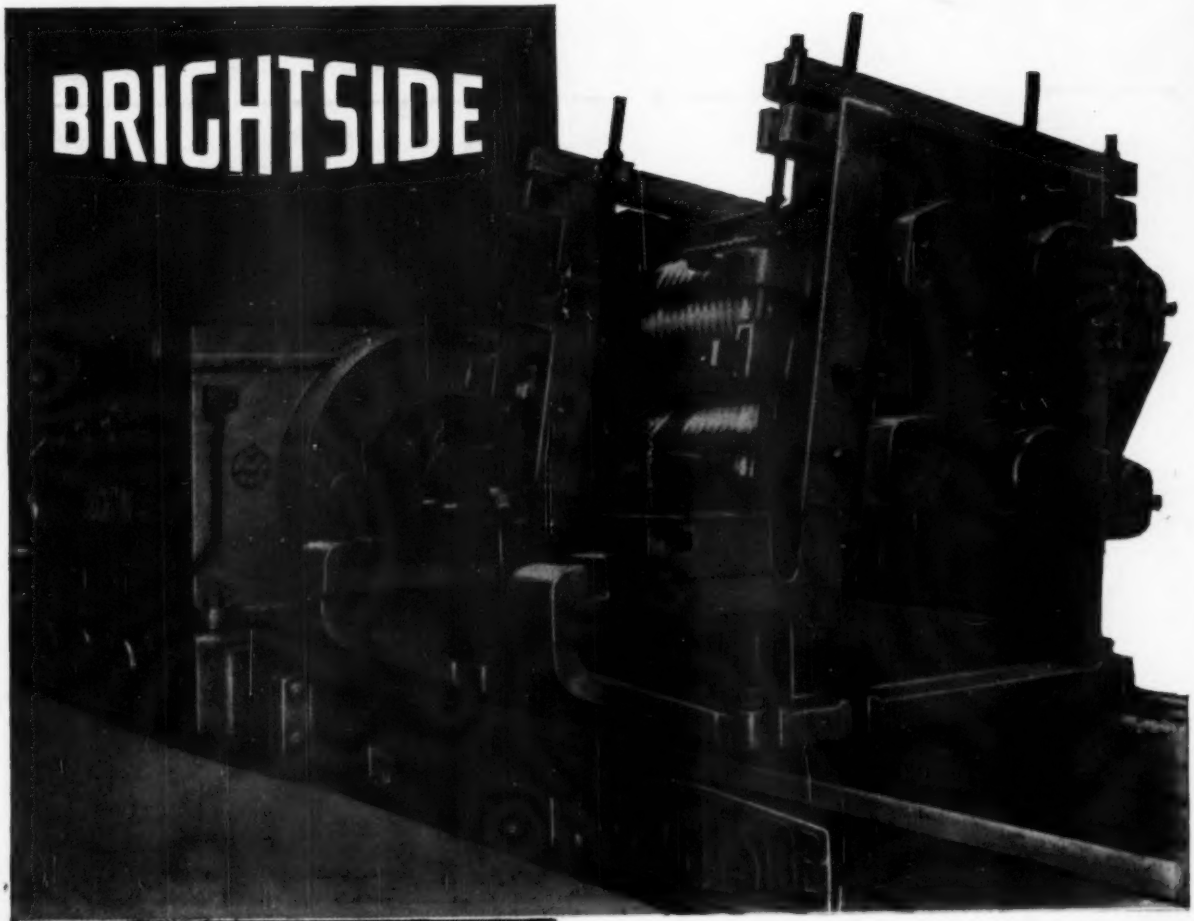

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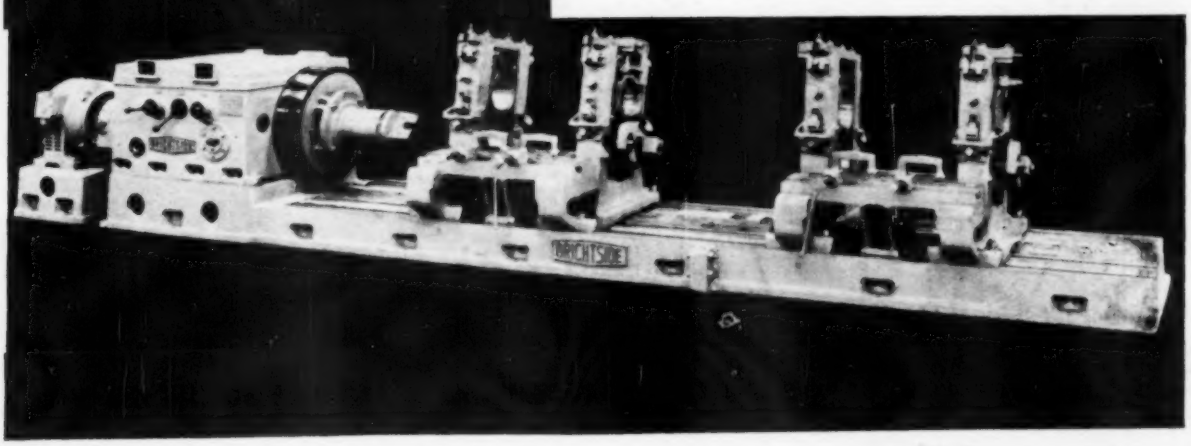
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